Fabrication for Nanotechnology

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Introduction

There is much discussion what nanotechnology really is: anything produced in the nanometer scale, manipulating atoms or small particles. Seeing the wide variety of materials and fabrication technologies one could better speak of nanotechnologies. In general the fabrication technologies could be divided into four groups as pictured in the following figure.

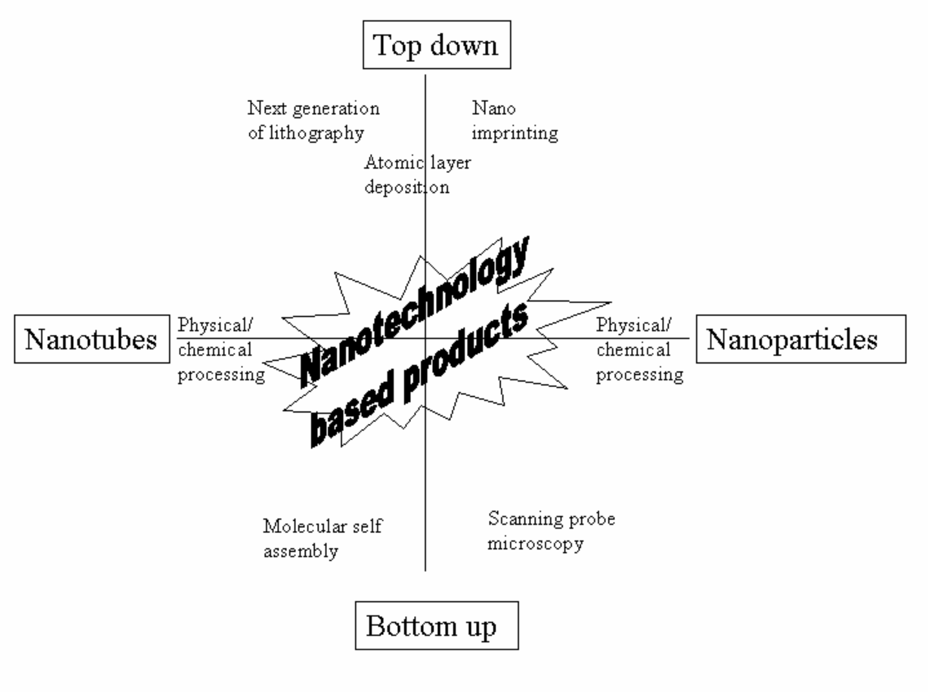

Figure 1: Nanotechnology fabrication methods

Top-down nanofabrication

The currently best developed area is the top-down nanofabrication with its base in the semiconductor industry. This industry is rapidly entering the sub nanometer area, and they need increasingly more accurate and therefore more expensive equipment. For semiconductor lithography nanoimprinting can offer a less complex process using more affordable equipment. The process uses a stamp instead of the more commonly used transparent mask. On a longer time frame, nanotubes are becoming of interest.
In the deposition area much attention is given to processes like Atomic Layer Deposition (ALD) and Molecular Layer Epitaxy, cyclic processes to make gap layers with high conformality and breakdown voltage. Basically the build up is layer by layer, whereby the layers can be varied according to need for specific layer properties.
Another deposition option is plasma deposition. The core is a DC magnetron which is used to sputter material into a liquid nitrogen cooled, high pressure aggregation/ drift region. The clusters (nanoparticles) formed in this region and are then channelled though apertures into the user's system and guided, as they have the properties of a beam, to a (cooled) substrate on which a layer is formed.

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Nanoparticle creation

The simplest process to create nanoparticles is the sol-gel process. Hereby mixing of ingredients dissolved in a liquid creates the initial particles. At a certain moment a surface active material is added to stop the growth. Hereafter the materials, still dispersed in liquid, can de transferred to the point of use.
Pyrogenic processing on the other hand is a process carried out in large expensive tools. It is used to create oxide particles by means of flame oxidation of metals, metalloids or their derivates in the gas phase.

A slightly more exotic process is called Controlled Detonation Synthesis and is used to create Nanoceramics and cubic carbon nanoparticles (Nanodiamonds). It involves the atomization of an explosion precursor material, immersed in specific gas medium. During the fly from the middle of reactor to the reactor walls atoms are clusterized and form nanoparticles.

Milling is a more traditional process. It can produce bulk metal powders of conventional, 10 – 1000 micron sizes, but with a nano-sized grain structure. A new variation of this process uses a mix of mechanical downsizing and chemical reactions. The chemical reactions are mechanically activated during milling, forming nanoparticles via a bottom up process. A ball mill acts as a low temperature chemical reactor. The particles are kept apart by salt matrix, which is removed through simple washing steps.

Nanotubes/fullerene production

The main challenges in nanotube fabrication are cost and purity. General a mix is created of several kinds of nanotubes. Depending on the process the balance between them can be shifted. Two broad ranges of processes can be distinguished: medium and high temperature processes.

The medium temperature category includes most of the catalytic processes like conventional Chemical Vapor Deposition (CVD), Plasma Enhanced CVD (PECVD), Hot Filament CVD and High Pressure Carbon Oxide CVD (HiPCo). The carbon source is a tightly controlled flow of a hydrocarbon gas (methane, carbon monoxide or acetylene). Metal catalyst such as Ni, Fe or Co are used to control the yield. Differences can appear in the methods used to decompose the gas. These methods are called “medium temperature” as the decomposition temperature is generally below 900°C. Medium temperature processes are particularly suitable for the synthesis of Multi Wall carbon nanotubes (MWNT).

The high temperature processes include laser ablation and electric arc discharge. In these methods, a composite target – made of graphite and a catalyst - is vaporized at high temperature (>1500°C). The carbon and the catalyst atomized, create a plume. In this zone carbon atoms combine to form carbon nanotubes. Like the medium temperature processes, the carbon nanotubes obtained are always contaminated with impurities such as amorphous carbon, residual metal catalyst and graphite nanoparticles. However, these methods provide very high structural quality material, monotype products (i.e. 100% of SWNT or MWNT) and they offer a strict control over the CNT diameter.

During the arc discharge process an arc is being created between a graphite cathode and a graphite anode. In the vapour phase carbon cluster are being created. These clusters are then cooled down and they condense on the surface of the graphite cathode. This deposit contains carbon nanotubes and impurities. By adding a catalyst (Co, Ni, Fe and Y powder), Single Wall NT are synthesized on the cathode.

Bottom up nanofabrication

The last part of the nanofabrication technologies is the bottom up fabrication involving the handling of individual atoms and molecules. The best known instrument here is the scanning probe microscope

(SPM). Advanced SPMs can be used to manipulate individual atoms. This is a rather slow fabrication technology of limited scope and efforts to create multiple probe systems meet high technological barriers. Nanoscribing is proposed as an alternative. Here a SW package transforms an AFM tool into a nano dispense unit. The achieved resolution is less then 10 nm. Probably the most promising area for this technology will be for the deposition of bioactive materials, which can not be deposited or structured via the harsh conditions of thin film processing. (I.e. writing of proteins patterns on gold surfaces)

Molecular self assembly can be achieved by using the ordered organization power of matter to produce homogenous monolayers with a high level of uniformity and reproducibility. It involves the dipping of a substrate into alternate aqueous solutions containing anionic and cationic materials, thereby creating subsequent layers on the substrate.

Far more futuristic is the idea to use molecules to create other molecules (nanofactories/molecular electronics).

Summary

Although many processes are discussed in this article the identified mainstream nanotechnologies are:

- arc combusting for nanotubes
- CVD for special nanotubes
- Molecular self assembly for monolayers
- Sol- gel process for nanolayers
- ALD for semiconductor isolating layers
- Nanoimprinting for thin film production
- Plasma processing for coatings and metal particles

The ultimate nanotechnology, the manipulation of atoms and molecules, has only been demonstrated yet in research environments.

Fabrication for nanotechnology

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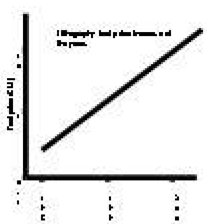
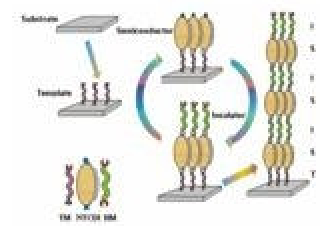
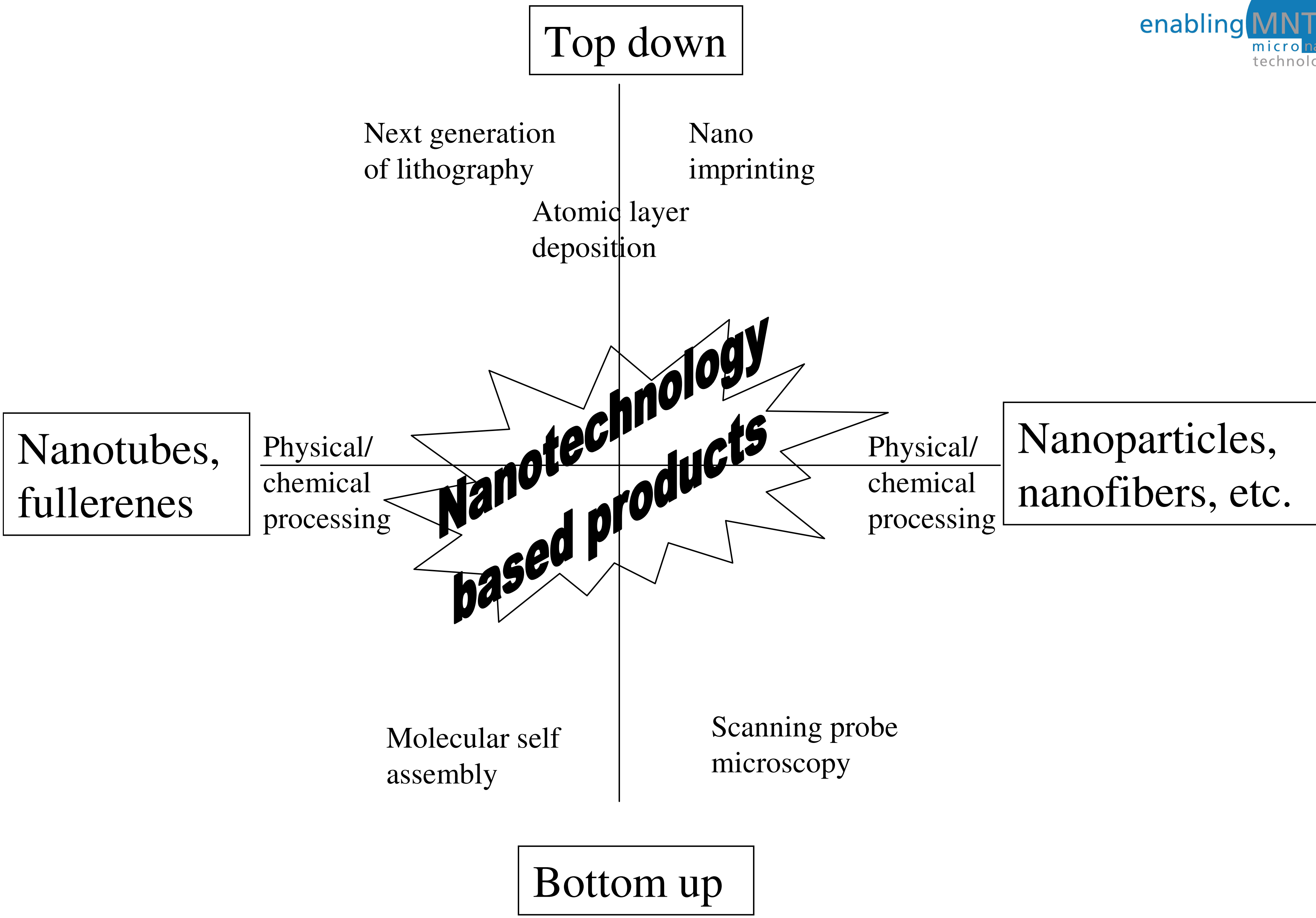




What is Nanotechnology?

- The atom by atom manufacture of material?
- Anything produced in the nanometer scale?
- Anything that is new and small?
- Anything new that leverages materials?
- Nanotechnology is molecular manufacturing?
- Many presented here – most define the field technologically

- **It’s not nanotechnology but nanotechnologies**


Top down
Next generation of lithography
Nano imprinting
Atomic layer deposition
Nanotubes, fullerenes
Physical/ chemical processing
Nanotechnology based products
Physical/ chemical processing
Nanoparticles, nanofibers, etc.
Molecular self assembly
Scanning probe microscopy
Bottom up

Contents

- Top-down nanofabrication
  - Lithography
    - nanoimprinting
  - Deposition
    - Atomic Layer Deposition
    - Molecular Epitaxy
    - Clusterbeam technology
    - Plasma technologies
- Nanoparticle creation
- Nanotubes/fullerene production
- Bottom up nanofabrication

Top-down

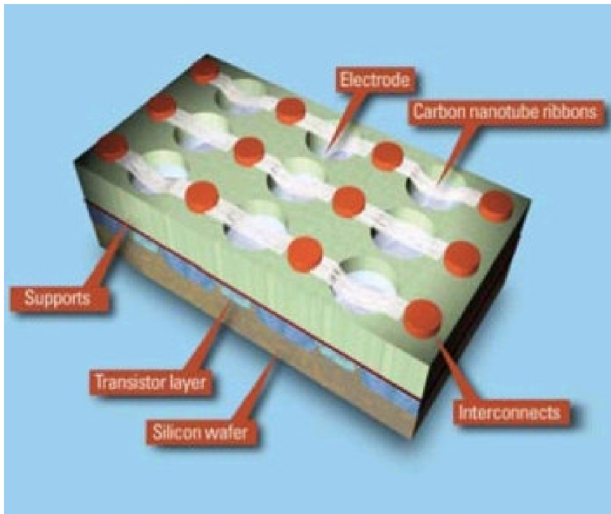
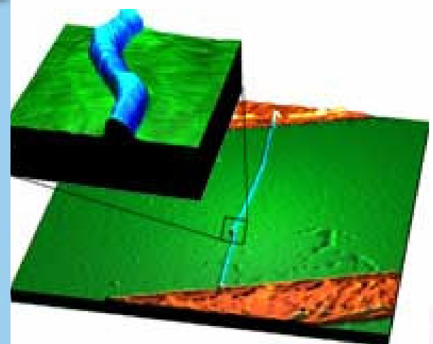


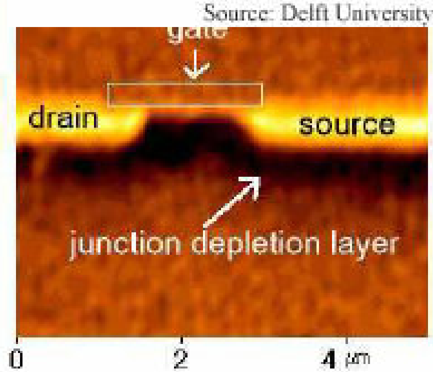
Image courtesy of Nantero

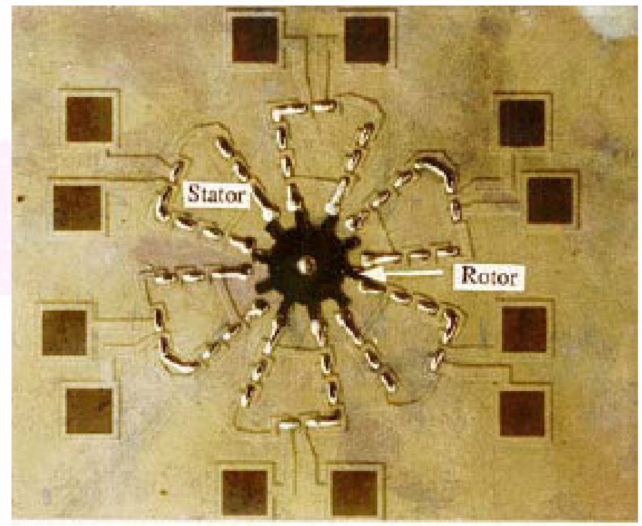
This illustration of Nantero's NRAM architecture shows how carbon nanotube ribbons deflect in response to an electric charge. Van der Waals forces hold the ribbon down, making the memory non-volatile.



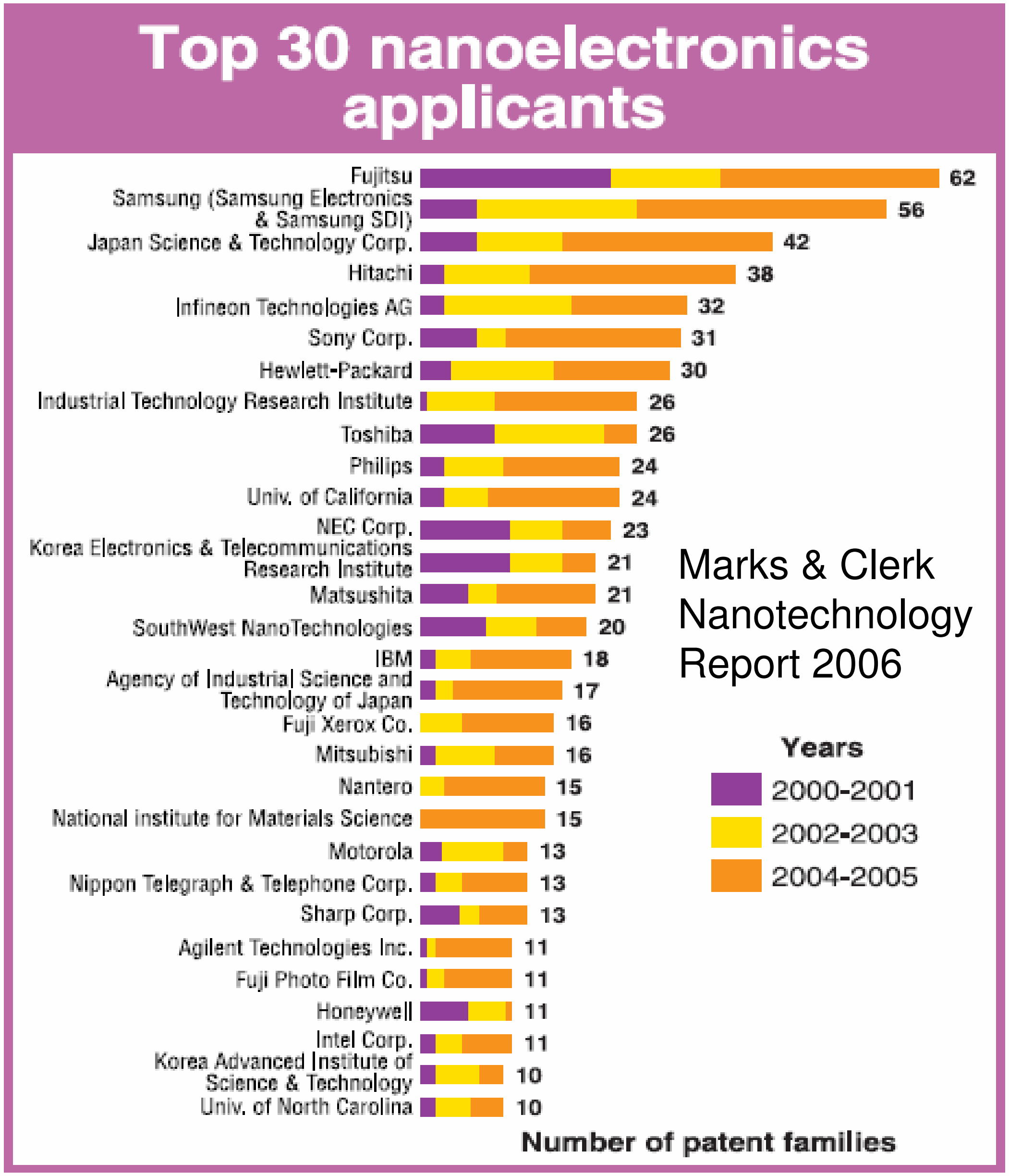
This image shows a closeup of a nanotube transistor section. The kinks in the nanotube act as electrodes that guide the flow of electrons.

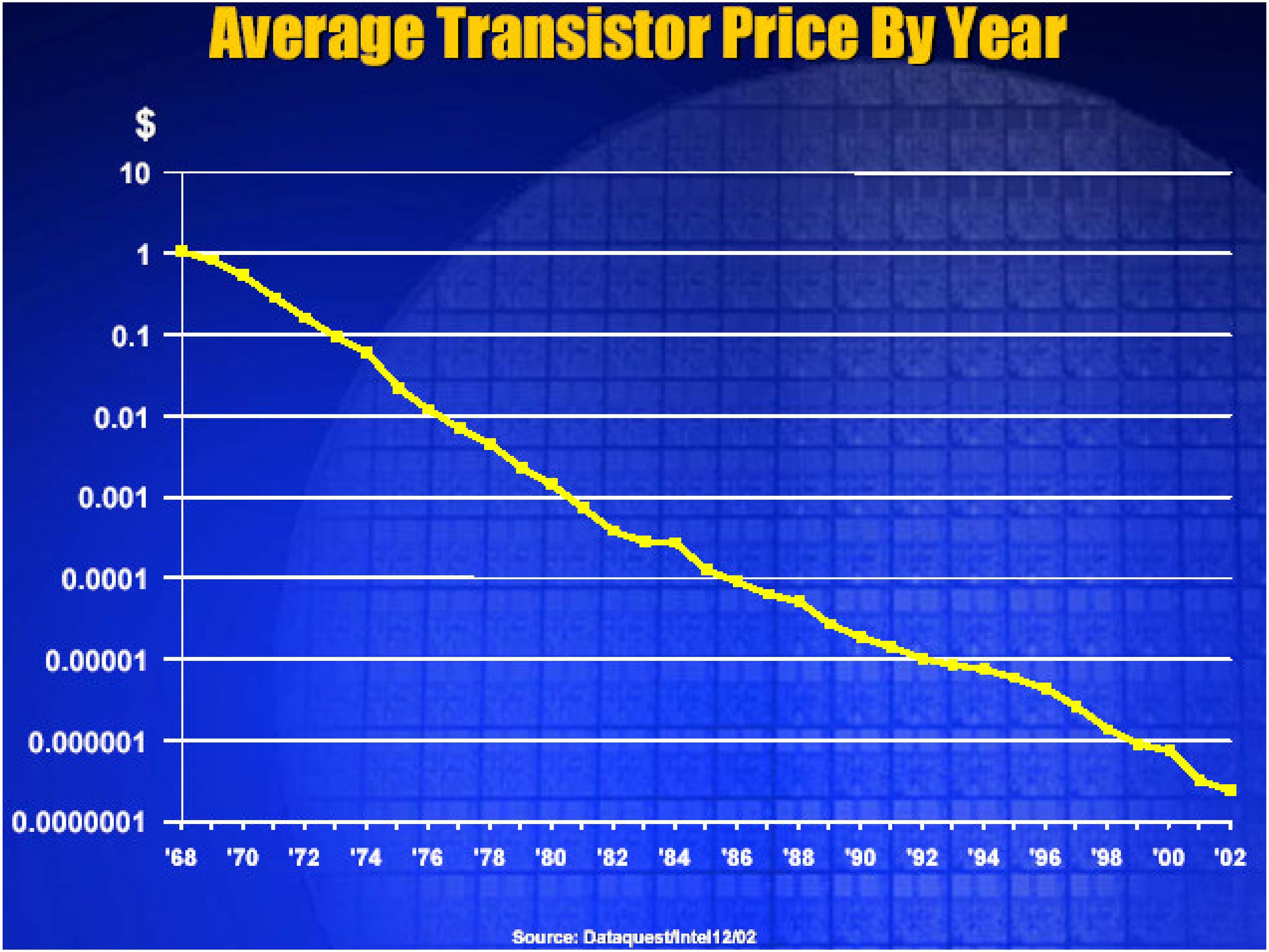
Source: Delft University





50µm


Top 30 nanoelectronics applicants
Fujitsu 62
Samsung (Samsung Electronics & Samsung SDI) 56
Japan Science & Technology Corp. 42
Hitachi 38
Infineon Technologies AG 32
Sony Corp. 31
Hewlett-Packard 30
Industrial Technology Research Institute 26
Toshiba 26
Philips 24
Univ. of California 24
NEC Corp. 23
Korea Electronics & Telecommunications Research Institute 21
Matsushita 21
SouthWest NanoTechnologies 20
IBM 18
Agency of Industrial Science and Technology of Japan 17
Fuji Xerox Co. 16
Mitsubishi 16
Nantero 15
National institute for Materials Science 15
Motorola 13
Nippon Telegraph & Telephone Corp. 13
Sharp Corp. 13
Agilent Technologies Inc. 11
Fuji Photo Film Co. 11
Honeywell 11
Intel Corp. 11
Korea Advanced Institute of Science & Technology 10
Univ. of North Carolina 10
Number of patent families
Marks & Clerk Nanotechnology Report 2006
Years
2000-2001
2002-2003
2004-2005



Average Transistor Price By Year
$
10
1
0.1
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0.001
0.0001
0.00001
0.000001
0.0000001
'68
'70
'72
'74
'76
'78
'80
'82
'84
'86
'88
'90
'92
'94
'96
'98
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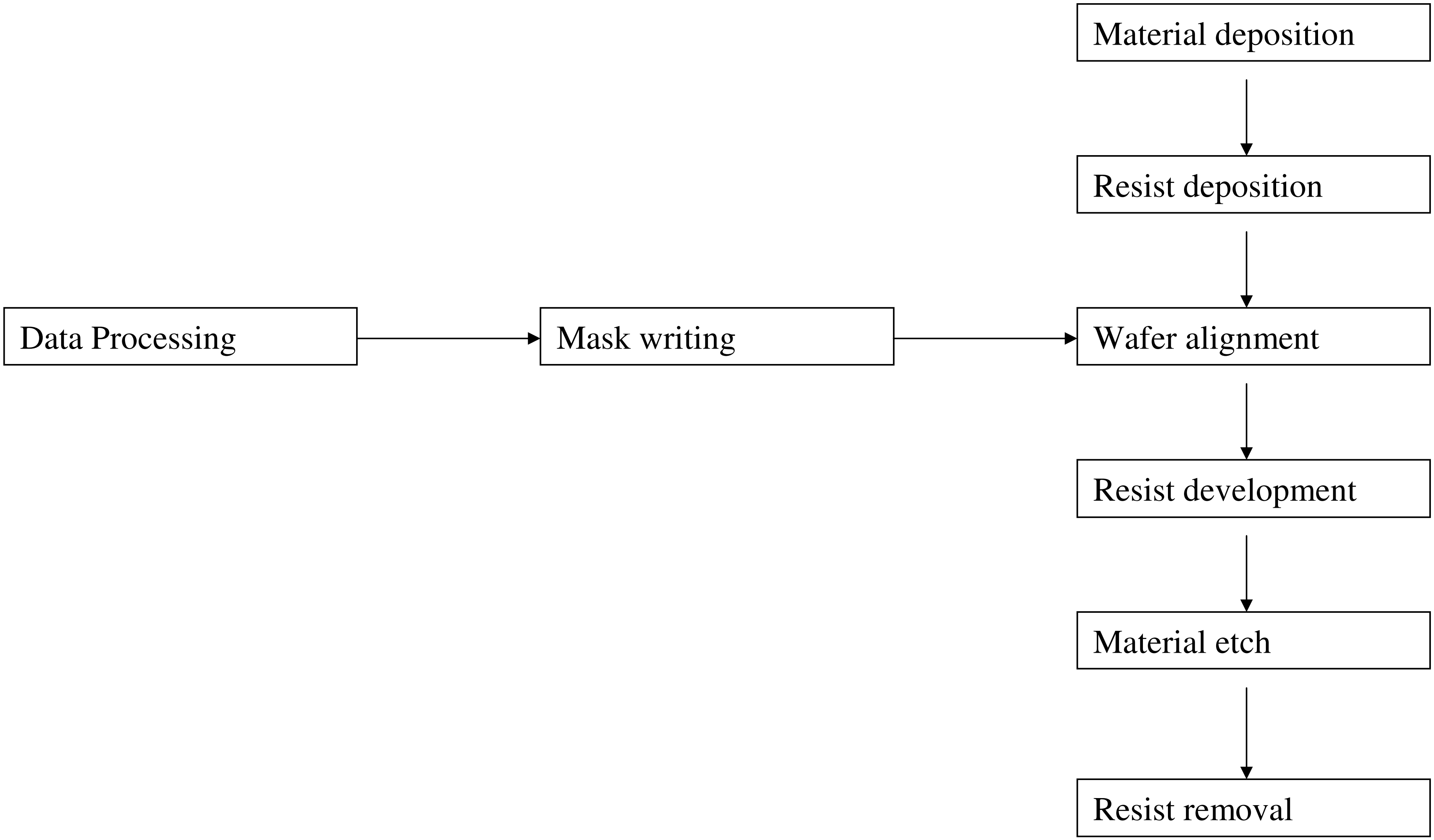
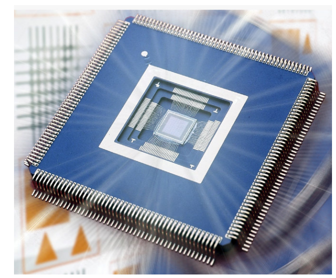
'02
Source: Dataquest/Intel 12/02

Lithography

- Optical going to Extreme UV?
- Optical replaced by e-beam?
- Optical replaced by Nanoimprint?


Material deposition
Resist deposition
Data Processing
Mask writing
Wafer alignment
Resist development
Material etch
Resist removal

Semiconductor processing

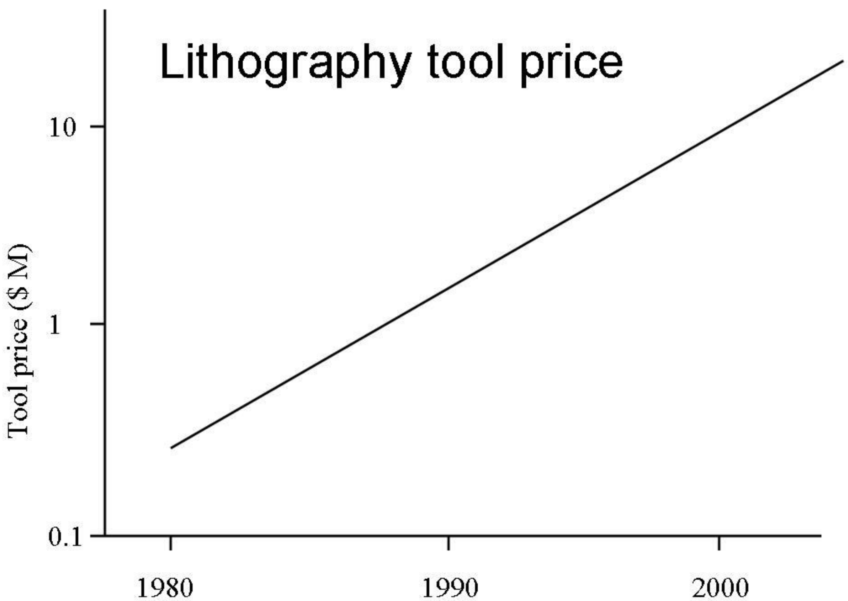


IBM scientists said they have created small, high-quality line patterns using deep-ultraviolet optical lithography.
The distinct and uniformly spaced ridges are only 29.9 nanometers wide, less than one-third the size of the 90-nanometer features now in mass production and below the 32 nanometers that industry consensus held as the limit for optical lithography techniques.

Semiconductor dilemma

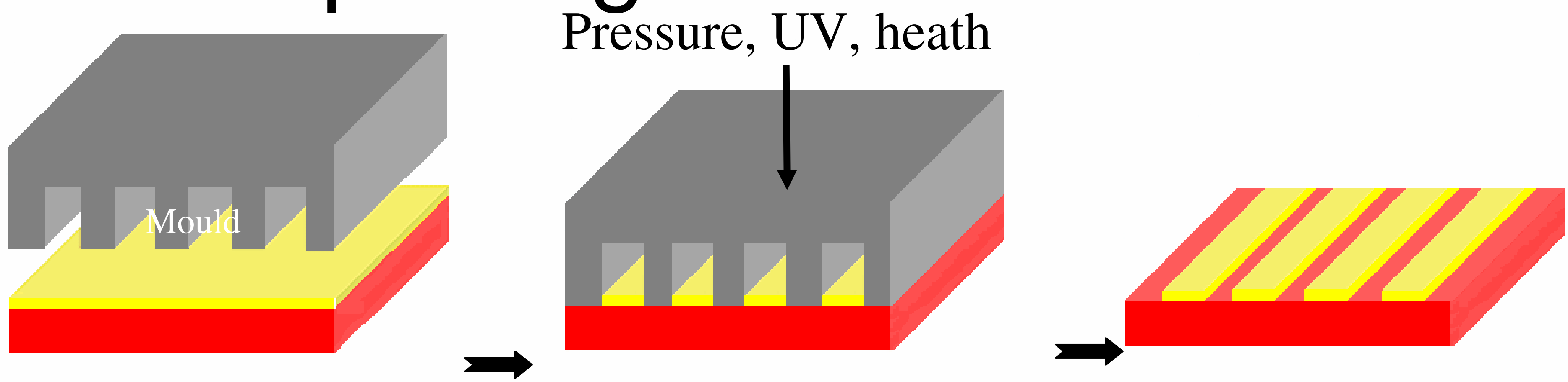
1) Lithography tools prices are rising fast

2) E-beam mask making is falling behind Moore's Law!!!

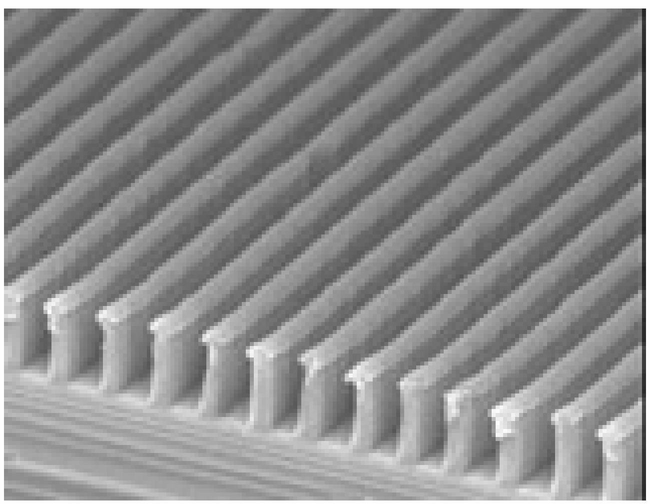


- There is a 25 percent increase in mask write times for every technology node.
- In fact, the average write times for a critical layer in a photomask has jumped to 24 hours, up from 5-to-6 hours in recent times!

Imprinting as alternative?



Substrate and mould — Imprinting process — Printed pattern

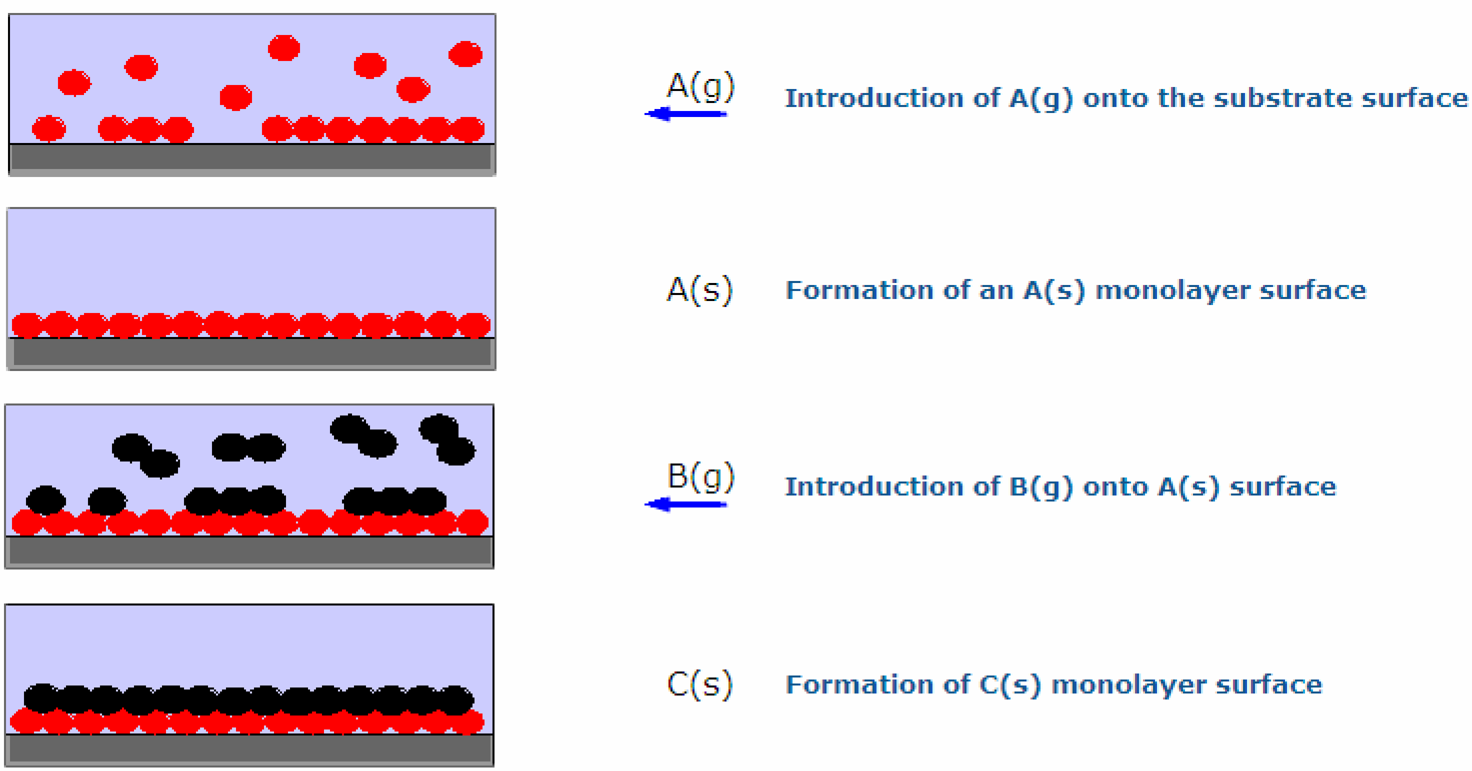


Promises to be a less complex process with more affordable equipment. Still needs (expensive and time consuming) creation of masks)

Semiconductor Deposition

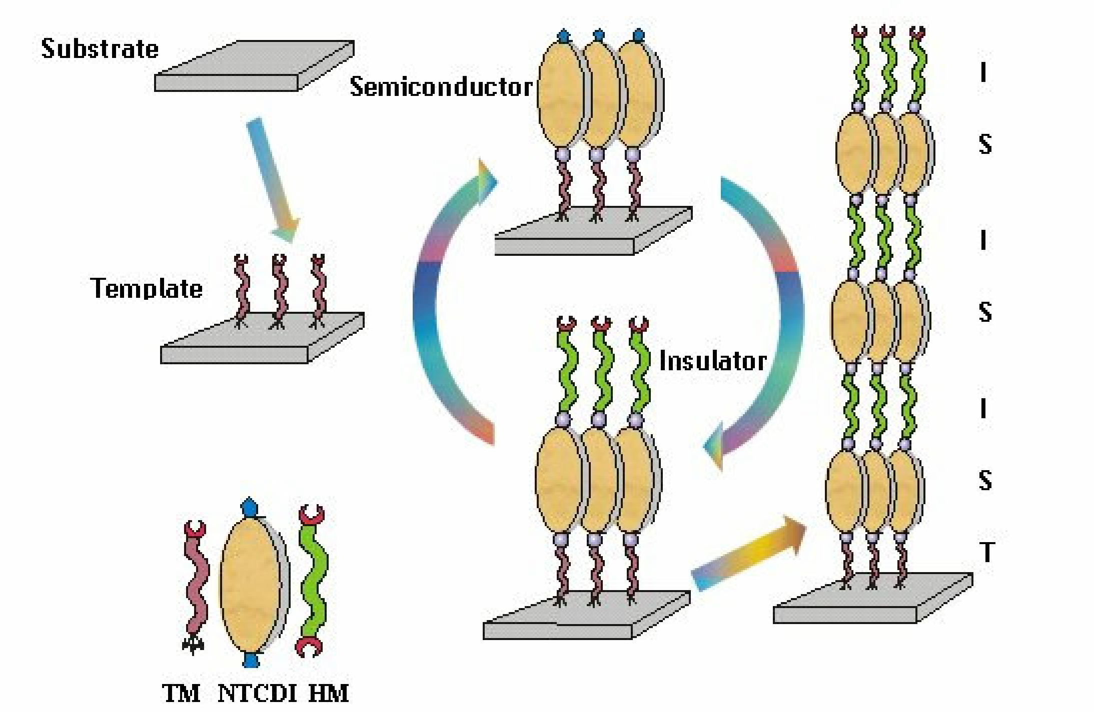
- Atomic layer deposition
- Plasma deposition

Atomic Layer Deposition



A cyclic MOCVD process to make gap layers with high conformality and breakdown voltage.

Molecular Layer Epitaxy



- 1) molecular template "hooks" upon a substrate, preparing the material to receive the first, semi-conducting layer.
- 2) an insulating layer of molecules is deposited.

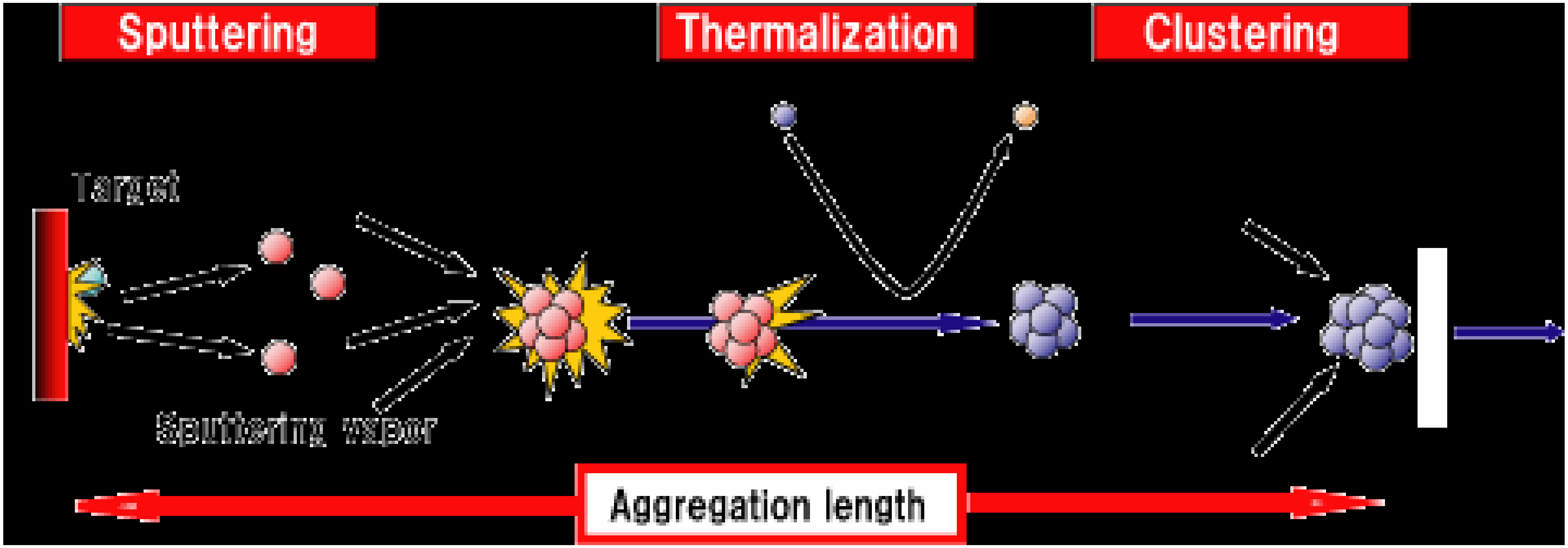
The process is repeated multiple times and in any combination required to attain the desired end result - the creation of tailor-made microelectronic devices.

Plasma deposition (1)

- The core is a DC magnetron which is used to sputter material into a liquid nitrogen cooled, high pressure aggregation/ drift region.
- The clusters form in this region and are then channeled though apertures into the user's system.
- A large percentage of the clusters generated by the source are ionized (typically 40% for Cu clusters). An ionized beam can be accelerated towards a substrate to form highly adherent and uniform coatings.
- High deposition rates: between <0.001nm/s and >0.5nm/s
- This technique can be used to form films on difficult materials, such as teflon.

Plasma deposition (2)

- A cluster (nanoparticle) can be treated as if they have the properties of beam in cluster beam technology.



Contents

- Top-down nanofabrication
- Nanoparticle and nanofiber creation
  - Nanoparticle
    - Wet chemistry
      - Sol-gel
    - Gas phase processing
      - Pyrogenic processing
      - Controlled Detonation Synthesis
    - Mechanical
      - Milling
  - Nanofibers
- Nanotubes/fullerene production
- Bottom up nanofabrication

Nanoparticle fabrication

Chemical

Biological

Mechanical:

- Milling
- Mechanochemical Processing
- High Velocity Deformation

Self assembly

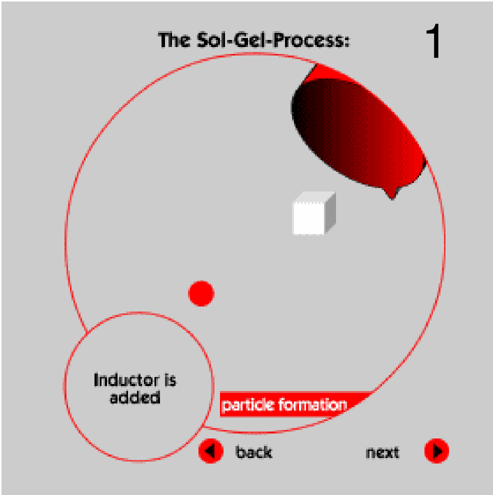
Gasphase processing:

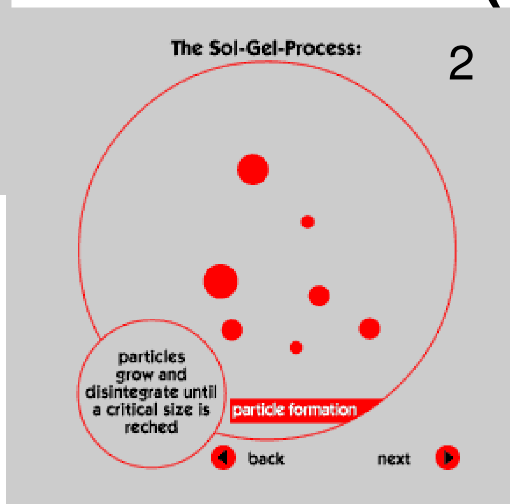
- Controlled Detonation
- Plasma Spray process
- Pyrogenic processing
- Flame Spray processing

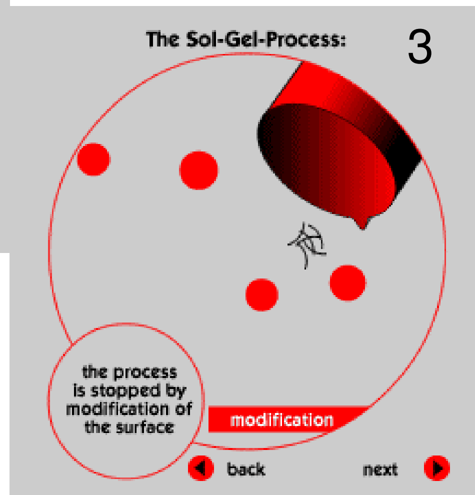
Liquid phase processing:

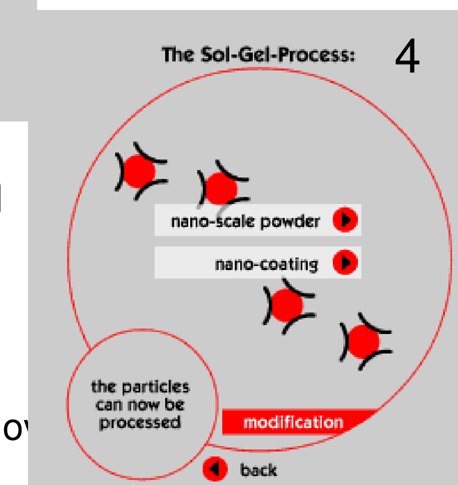
- Sol-gel process

Sol-gel process (Nanogate)









Improvement: Use CO_2 instead of H_2O!

1) Mixing of ingredients

2) Growth of particles

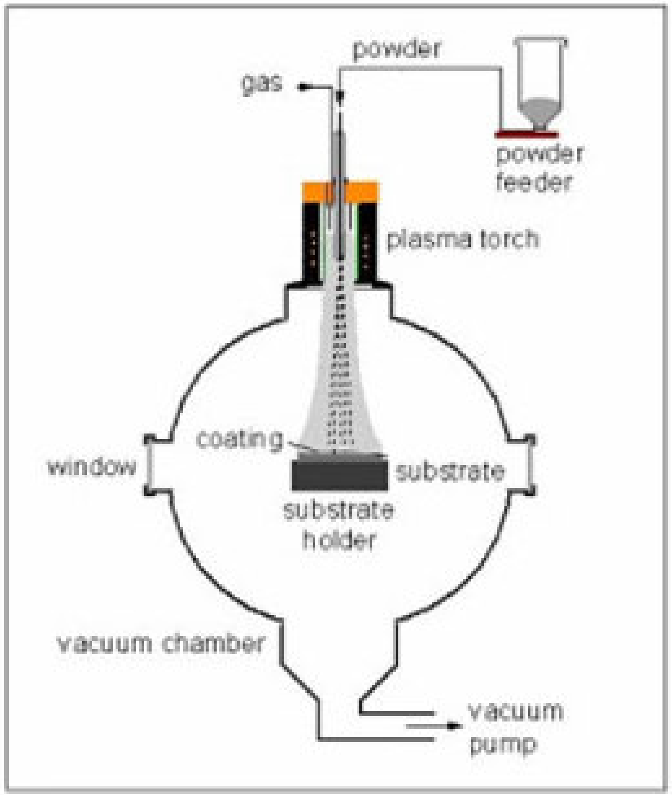
3) Adding surface active material to stop growth

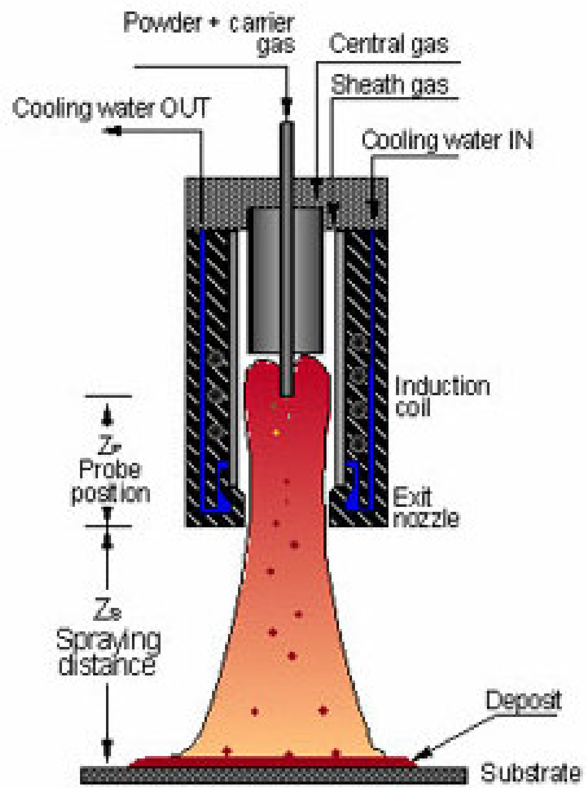
4) Transfer to point of use

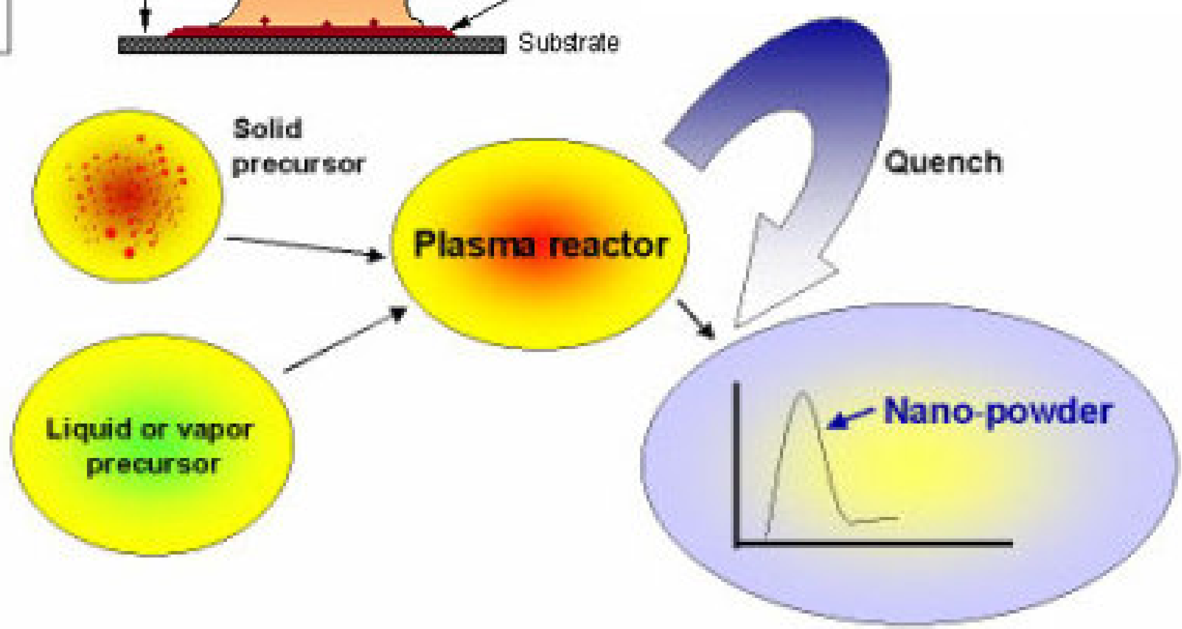
Pyrogenic processing

- Pyrogenic processing is the formation of particles by means of flame oxidation of metals, metalloids or their derivates in the gas phase.
- The flame with temperatures from 1000℃ up to 2400℃ provides the energy to evaporate the precursors and to drive the chemical reactions.
- With extremely short residence times of 10 to 100 ms in the highest temperature region, this zone is crucial for the formation of the primary particles.
- Downstream the size and the morphology of the aggregates are hardly influenced

Plasma processing







Solution Plasma Spray process (Inframat)

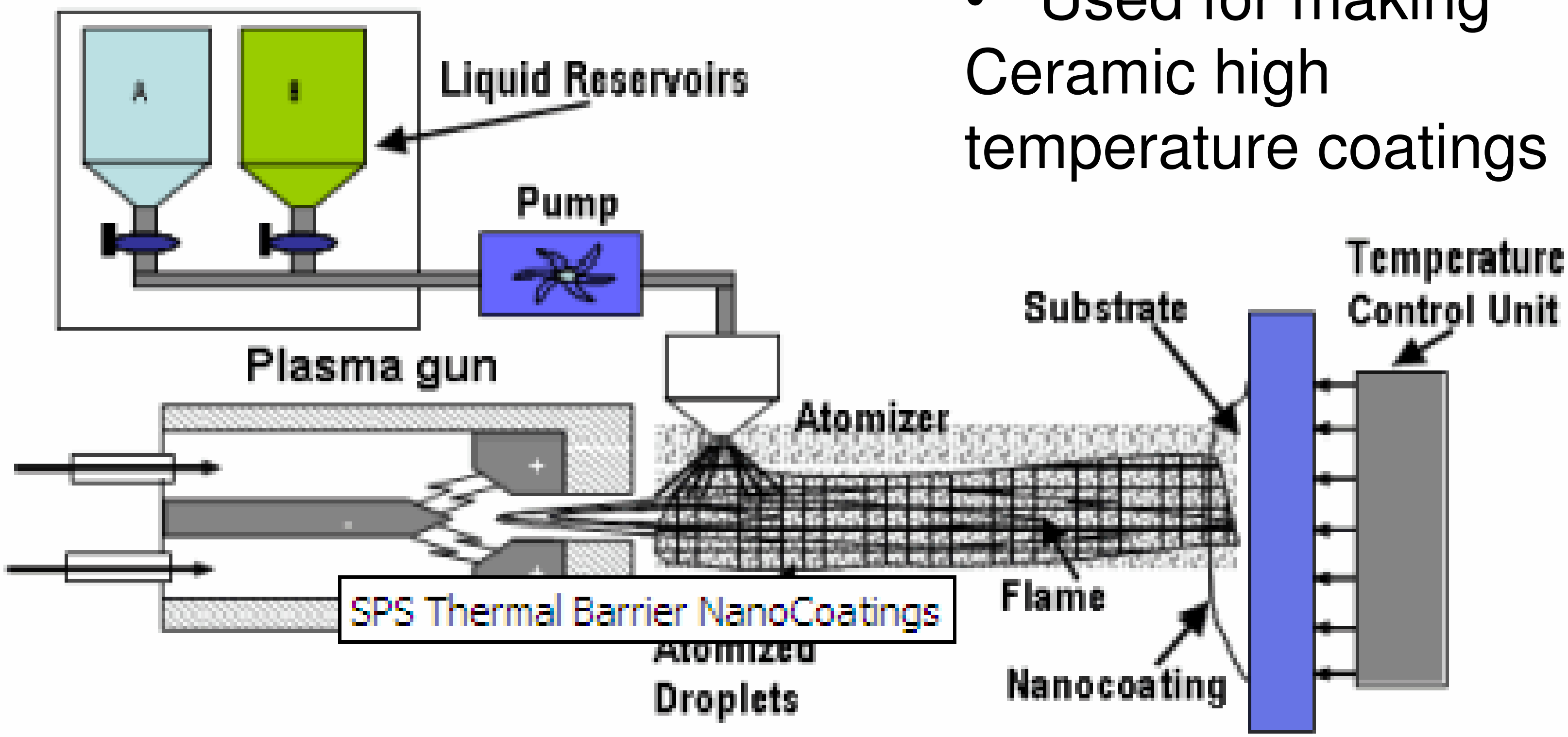
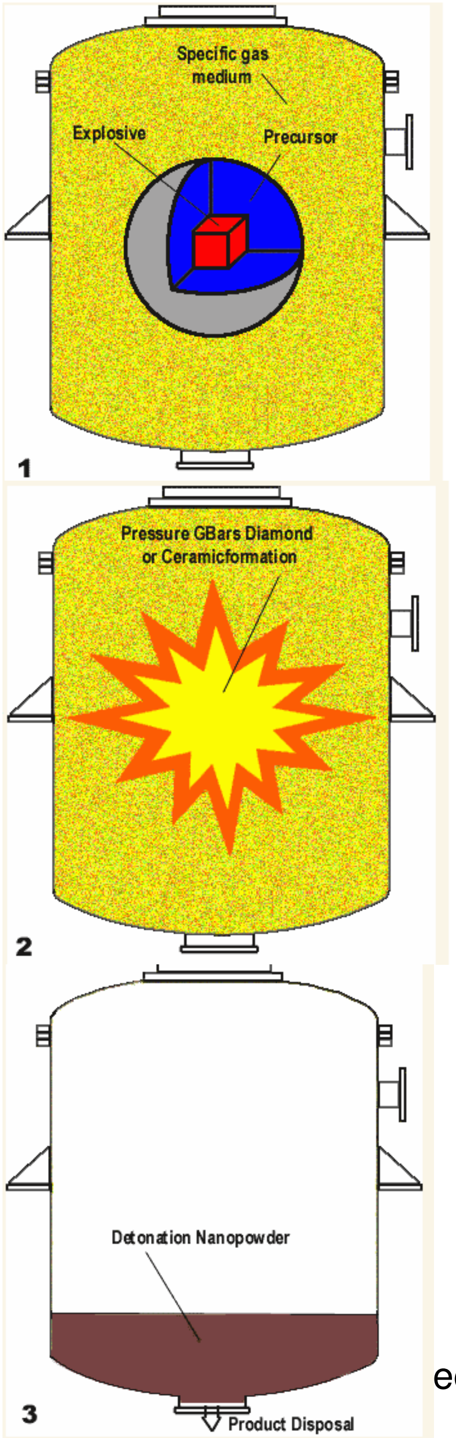


Fig. 3. Solution Plasma Spray Process

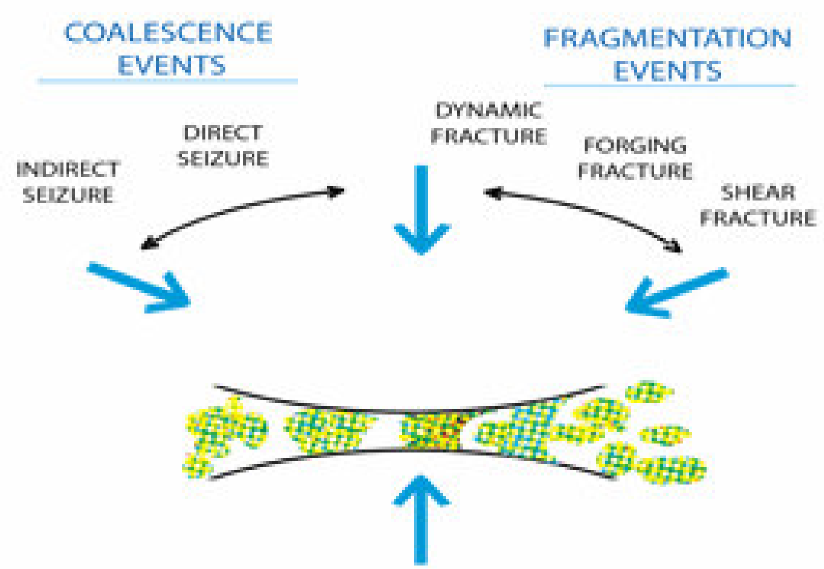
- Used for making Ceramic high temperature coatings



Controlled Detonation Synthesis (PlasmaChem)

- At explosion precursor material, immersed in specific gas medium, is atomized.
- During the fly from the middle of reactor to the reactor walls atoms are clusterized and form nanoparticles.
- During whole synthesis process the high pressure is maintained enabling formation of Nanoceramics and cubic carbon nanoparticles (Nanodiamonds).

Milling



High-energy ball milling has been applied, beyond mechanical alloying, to the synthesis of a variety of materials either starting from:

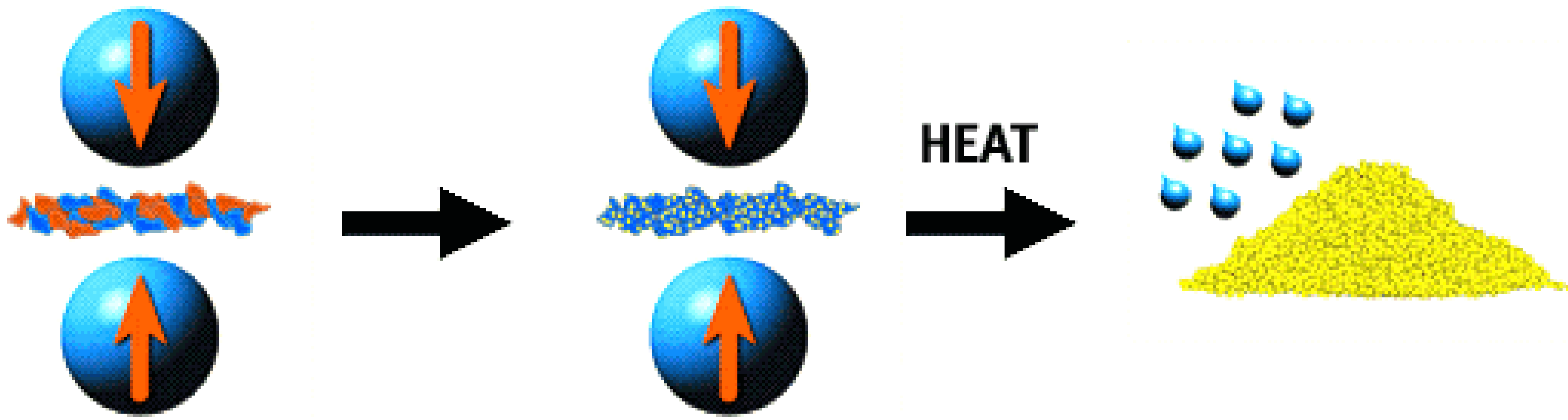
- elemental powders for most carbides, silicides and intermetallic compounds
- from compounds for (metal)carbides and metaloxides

Particle sizes are controlled either by in-process or post-process steps; average particle sizes down to the micron range can be obtained.

Mechano-chemical Processing

- Mechano-chemical processing is a low cost method of manufacturing a wide range of high quality nanopowders in a conventional ball mill.
- This is not just a grinding process.
- Chemical reactions are mechanically activated at the nanoscale during milling, forming nanoparticles via a bottom up process.

Mechanochemical Processing Advanced Nano

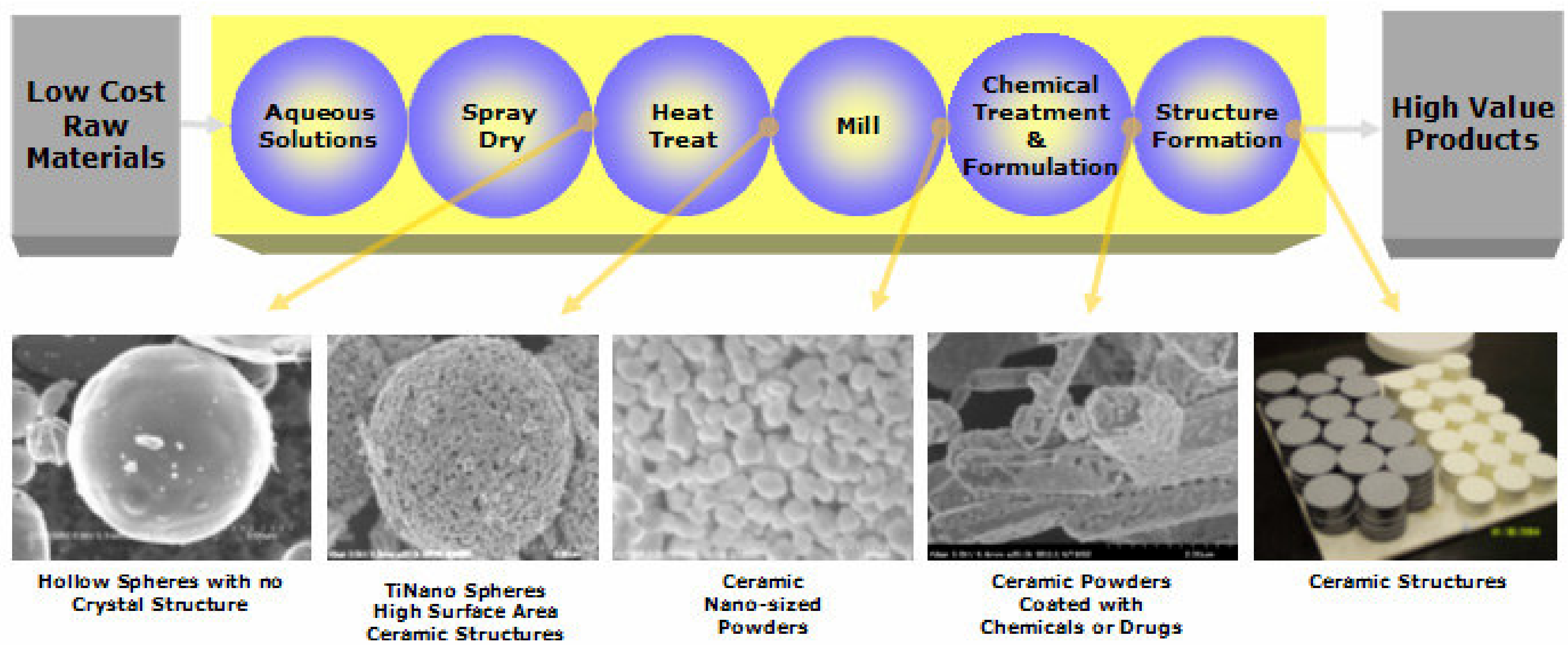


Ball mill acts as a low temperature chemical reactor. Reaction process results from focal heat and pressure at contact surface.

Chemical reactions occur at nanoscale . Particles are kept apart by salt matrix. Low temperature enables controlled particle formation

Reaction product is heat treated. Solid phase chemistry prevents particles from agglomeration. Salt removed through simple washing steps.

Altair spray hydrolysis process



Often combination of processes (Altair)

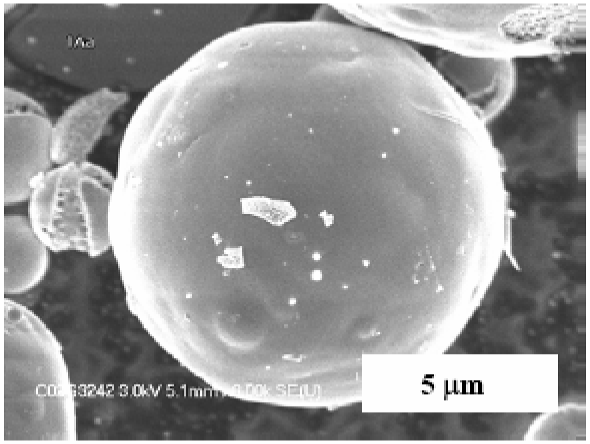


Figure 5a: Spray-hydrolysis product

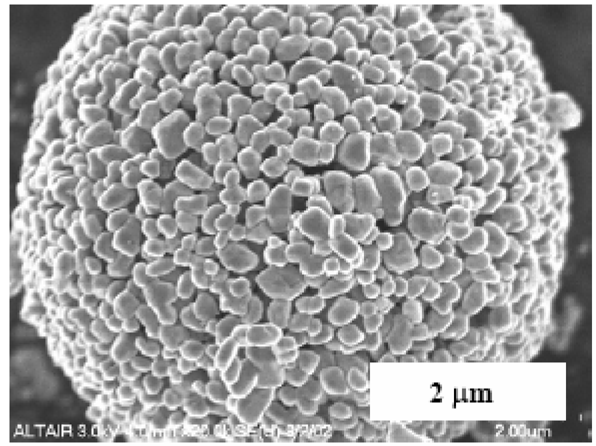
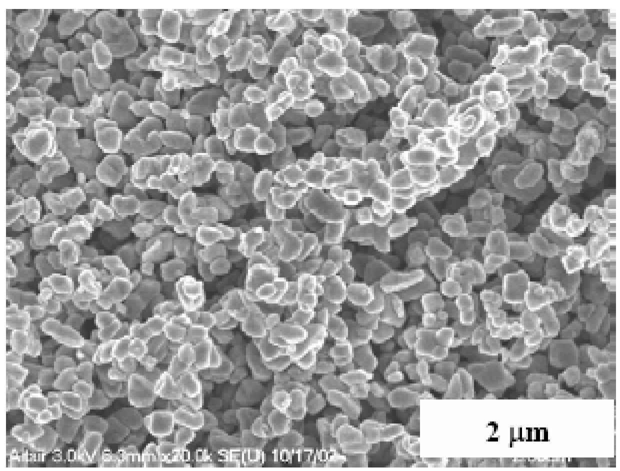


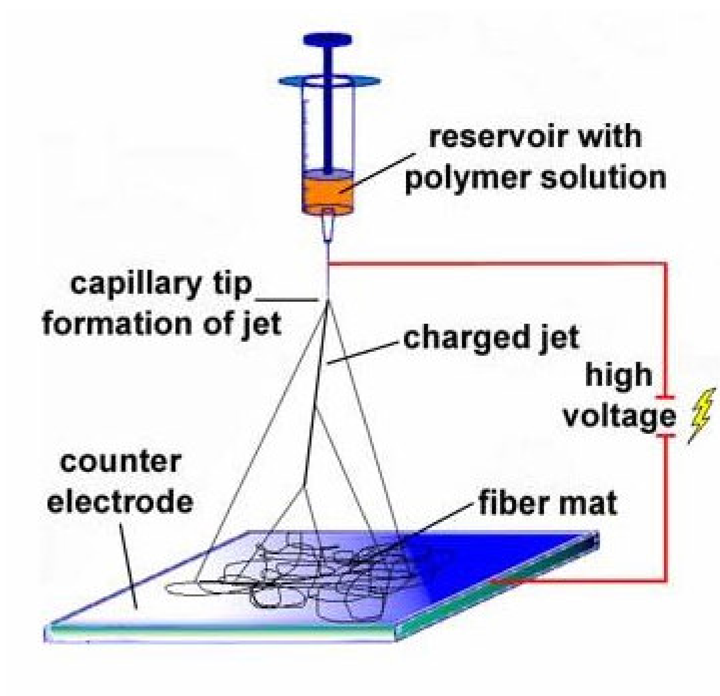
Figure 5b: Calcination product



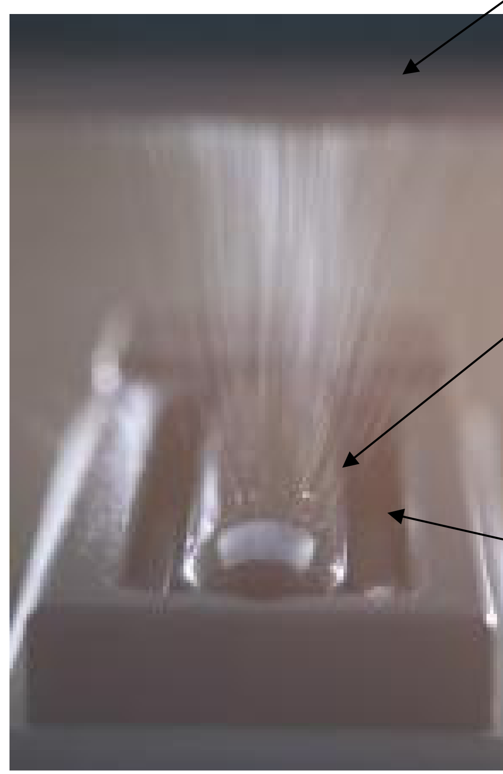
After milling

Nanofibers

Conventional (low volume production)



Elmarco process



Nanofibers

Thin film on rotating cylinder

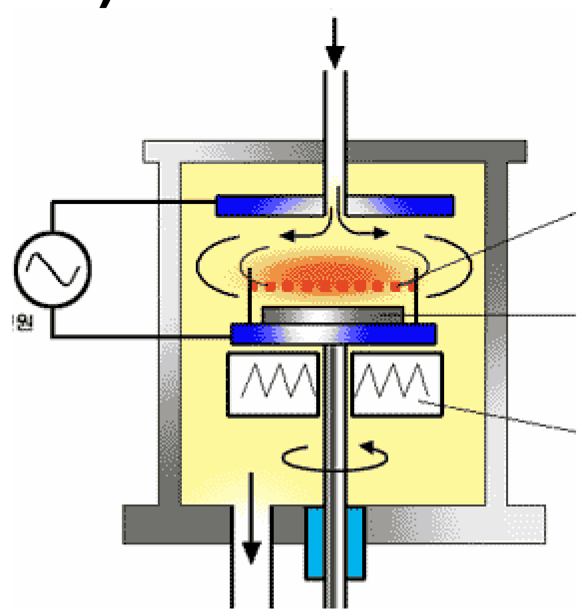
Solution containing polymer

Contents

Nanotube fabrication status

- Hottest area in nanotech!
- Several production technologies proposed
- Main challenges in nanotube fabrication:
  - nanotube cost
  - polydispersity of nanotube types, i.e. different (n,m) structures, and
  - dispersion and handling / orientation
- Basics about nanotube growth not quit clear.

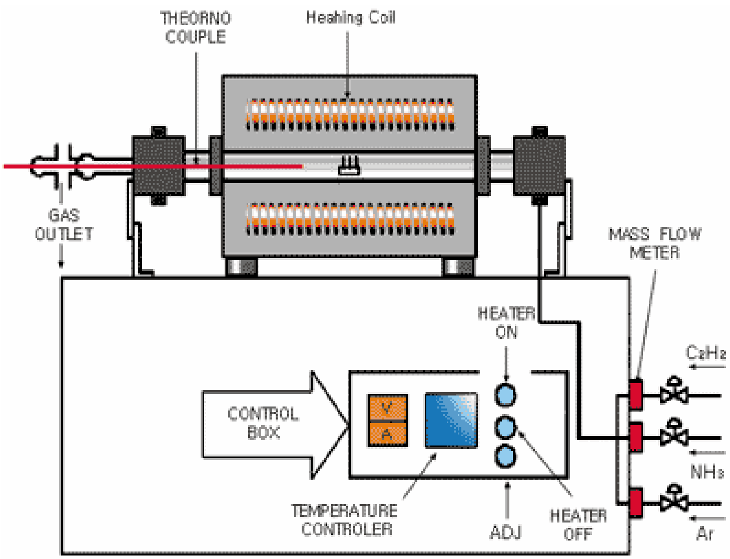
Plasma Enhanced Chemical Vapor Deposition (CEVP)

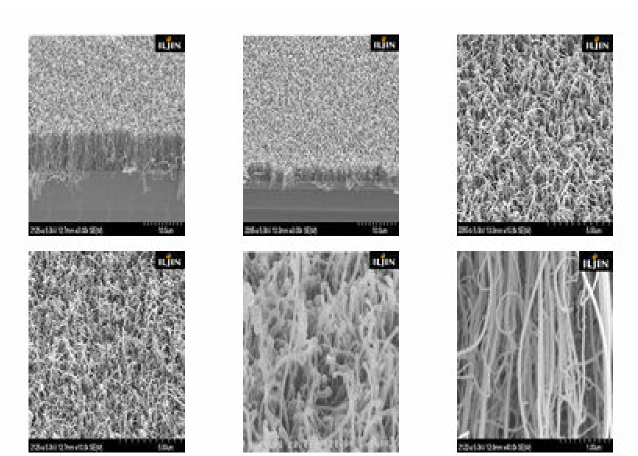


- Plasma-enhanced chemical vapour deposition process that is optimised for the growth of carbon nanotubes with highly controlled properties such as density, length and position.
- The integrated thermal control system maintains the work area substrate at room temperatures during processing, allowing carbon nanotube materials to be grown vertically and horizontally with precision

- Even deposition on highly heat sensitive materials such as plastic or metallised paper is possible.
- The high degree of thermal control offered by this tool means that the particles used to catalyse material growth remain at their original size, facilitating both precise and repeatable processing.

Thermal Chemical Vapor Deposition (ILJIN Nanotech)





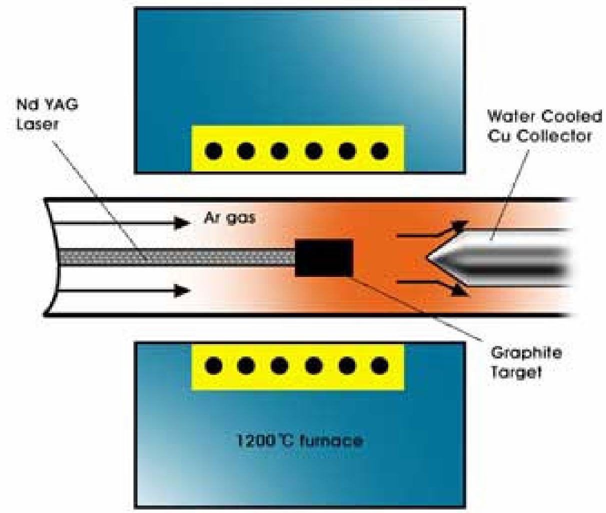
- Fe catalytic metal is patterned on a substrate, carbon nanotubes can be grown in accordance with the forms of catalytic metals.
- Reaction gas is supplied in one end of the apparatus, and gas outlet in the other.
- Vertically aligned carbon nanotubes are bound together as bundles by Van der Waals force.

CoMoCAT (Southwest Nanotechnologies)

- Catalytic method
- SWNT are grown by CO decomposition into C and CO_2 at 700-950ºC in flow of pure CO at a total pressure that typically ranges from 1 to 10 atm.
- Selectivity towards SWNT better than 90 %
- Carbon content is greater than 90% Two main types of semiconducting SWNT are present; mainly (6,5) + (7,5). The average diameter is 0.8nm, +- 0.1nm.

Laser ablation

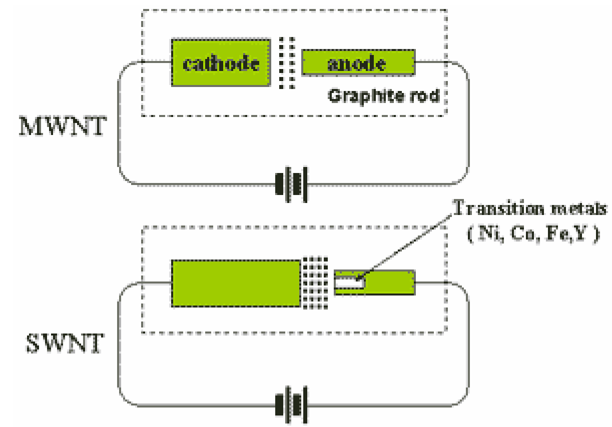
- High temperature methods include laser ablation (pulsed or not), solar method and electric arc discharge. In all these methods, a composite target – made of graphite and a catalyst - is vaporized at high temperature (>1500℃).
- The carbon and the catalyst atomized, create a plume where the density of each component is one of the key point. In this zone carbon atoms combine to form carbon nanotubes.



- Like the medium temperature processes, the carbon nanotubes obtained are always contaminated with impurities such as amorphous carbon, residual metal catalyst and graphite nanoparticles.
- However, these conventional methods provide very high structural quality material, monotype products (i.e. 100% of SWNT or MWNT) and they offer a strict control over the CNT diameter.

Arc discharge

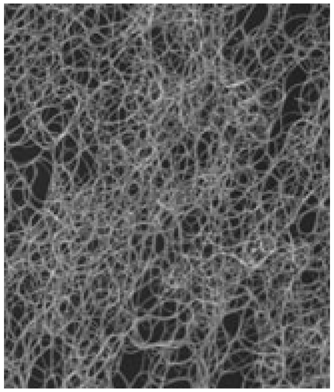
- An arc is being created between a graphite cathode and a graphite anode, thus moving and colliding a large quantity of electrons into the anode rod.
- The carbon clusters are then cooled down and they condense on the surface of the graphite cathode.
- The deposit contains carbon nanotubes and impurities.
- By adding a catalyst (Co, Ni, Fe and Y powder), Single Wall Nanotubes are synthesized on the cathode.
- By working on the gas parameters, on the energy feed stock and on the catalyst, the arc discharge method can be optimized in order to produce high performance Single Wall nanotubes.

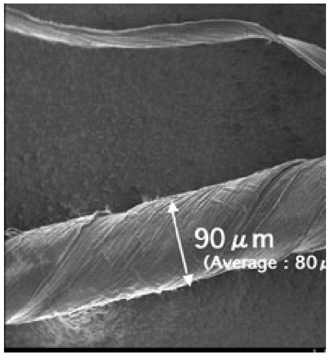


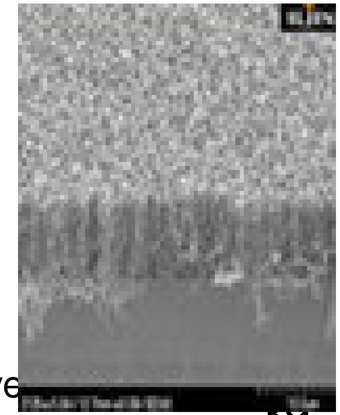
Purification of nanotubes

- In gas phase
- In liquid phase
  - Remove large (carbon) particles
  - Dissolution to remove fullerenes and catalyst particles
  - Centrifugal separation (concentration)
  - Microfiltration
  - Chromatography
- Intercalation

Orientation of nanotubes





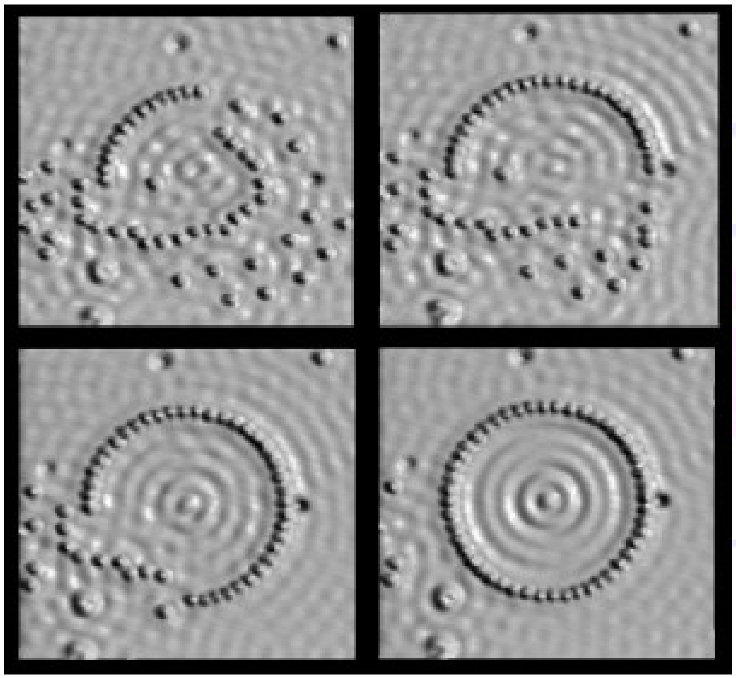


- Bulk (to be used as filler material etc)
- Spinning (high strength wire)
- Aligned (electron emitters in flat-panel displays)

Contents

- Top-down nanofabrication
- Nanoparticle creation
- Nanotubes/fullerene production
- Nanofibers
- Bottom up nanofabrication
  - Scanning Probe Microscopy
  - Nanoscribing
  - Molecular Self Assembly

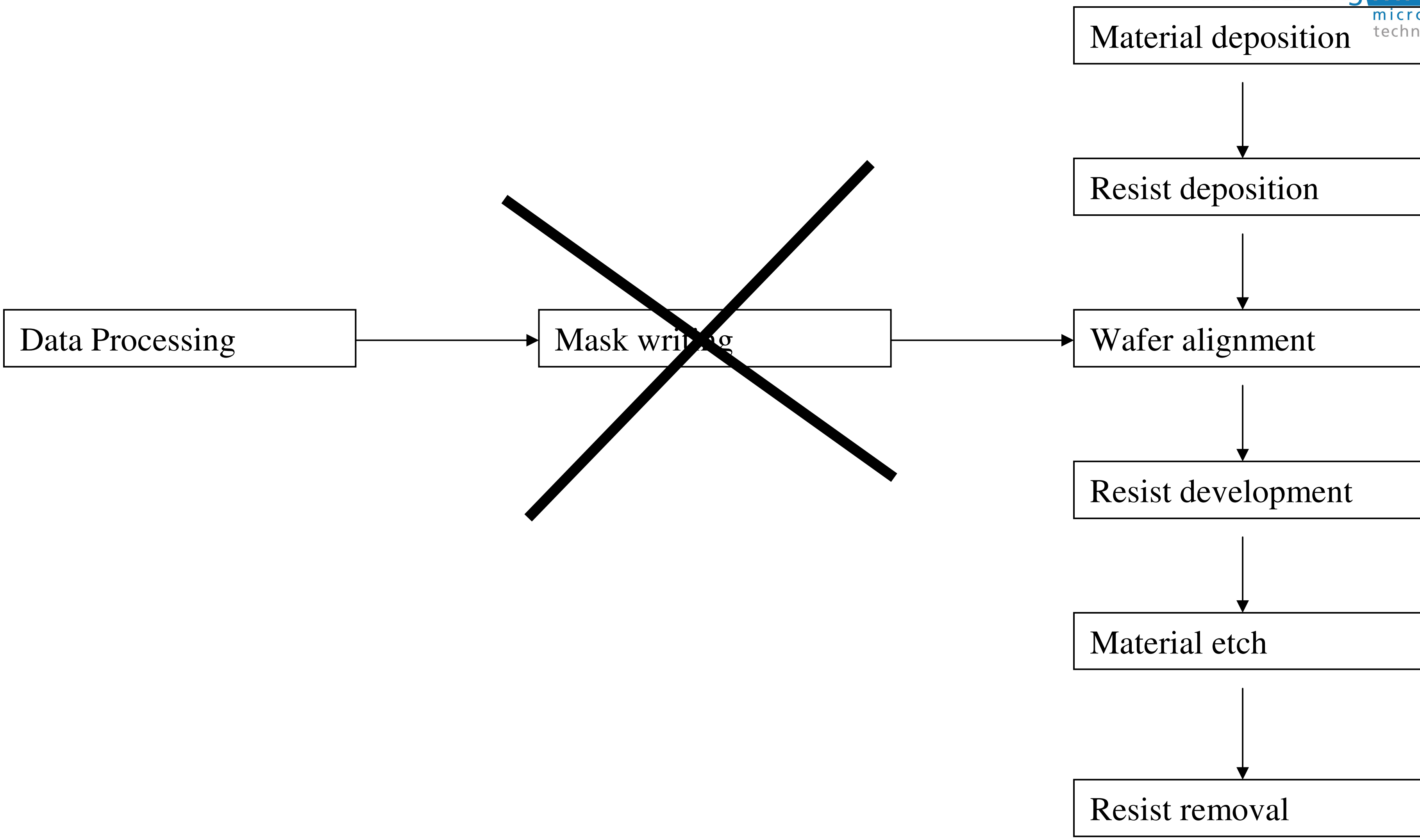
Bottom-up



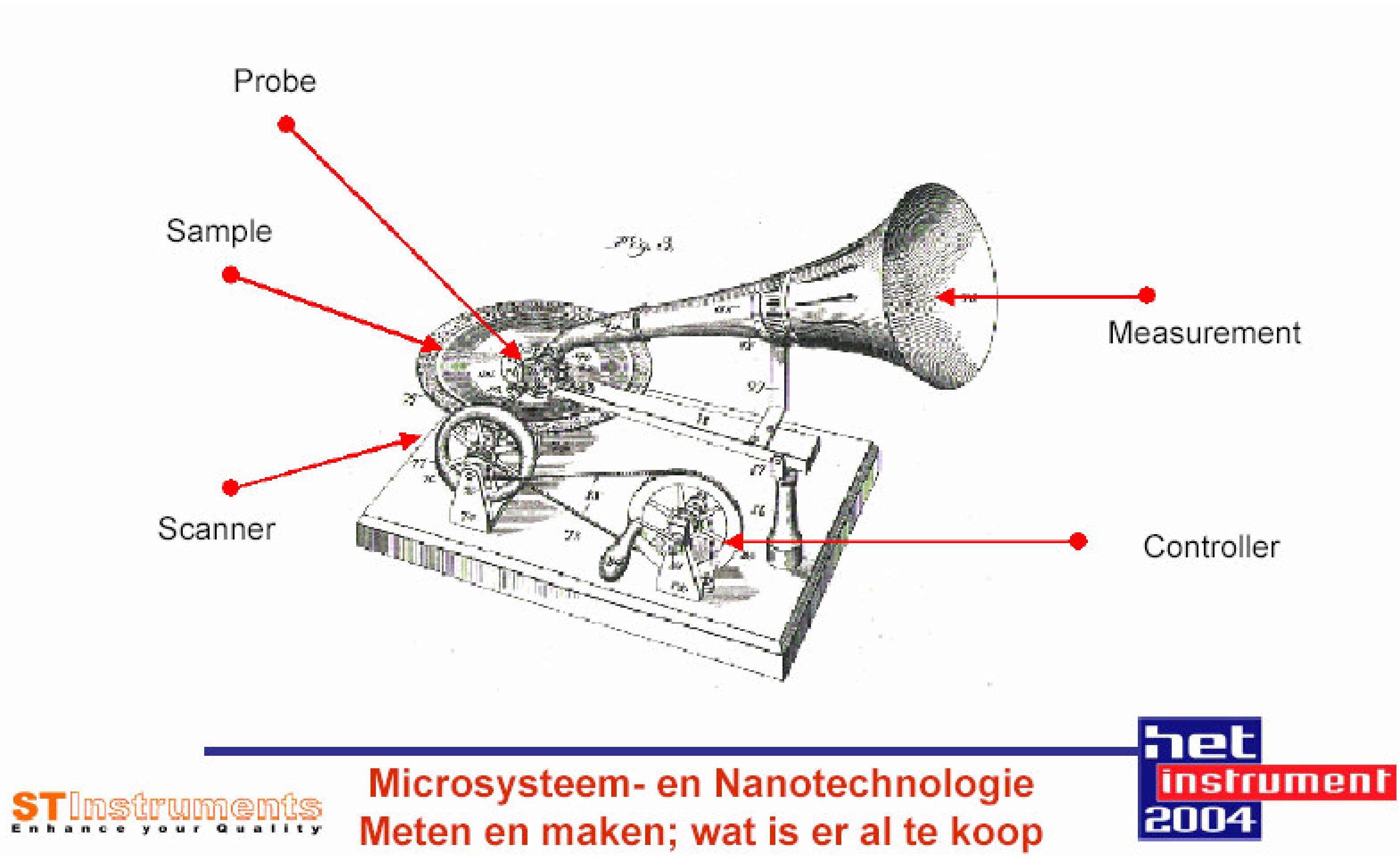
The making of a Circular Corral of Iron Atoms on a Copper Surface (111 oriented)
(copyright IBM)

Bottom-Up
Nano Manufacturing

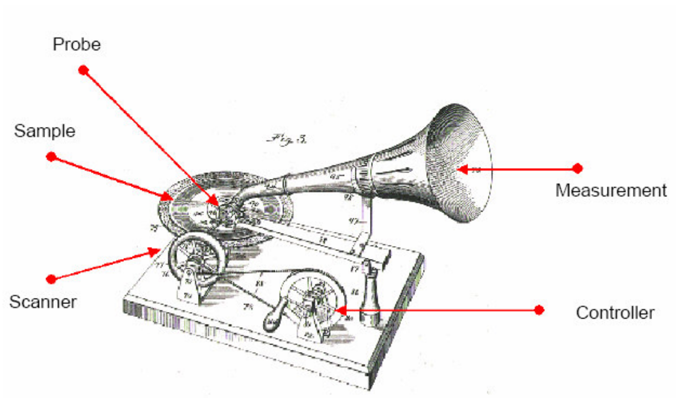
- The third wave of Micro Manufacturing
- Atom by atom manufacture
- Self assembled
- A material, chemical or biological based process
- The chemical and steel industry has utilized nano reactions in their processes for centuries
- Revitalization of chemistry and biochemistry

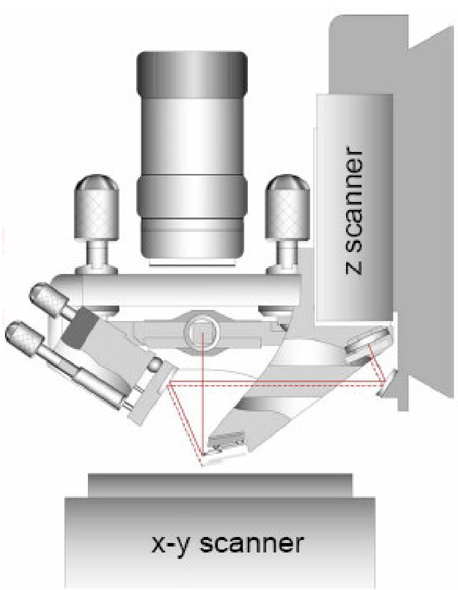

Material deposition
Resist deposition
Data Processing
Mask writing
Wafer alignment
Resist development
Material etch
Resist removal

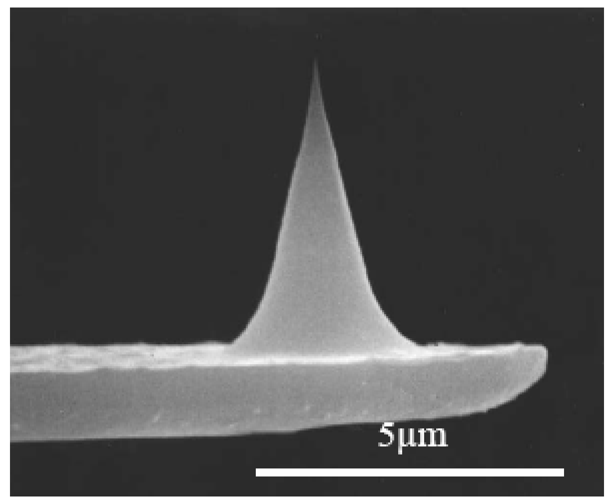
Scanning Probe Microscope (1)



Scanning Probe Microscope (2)

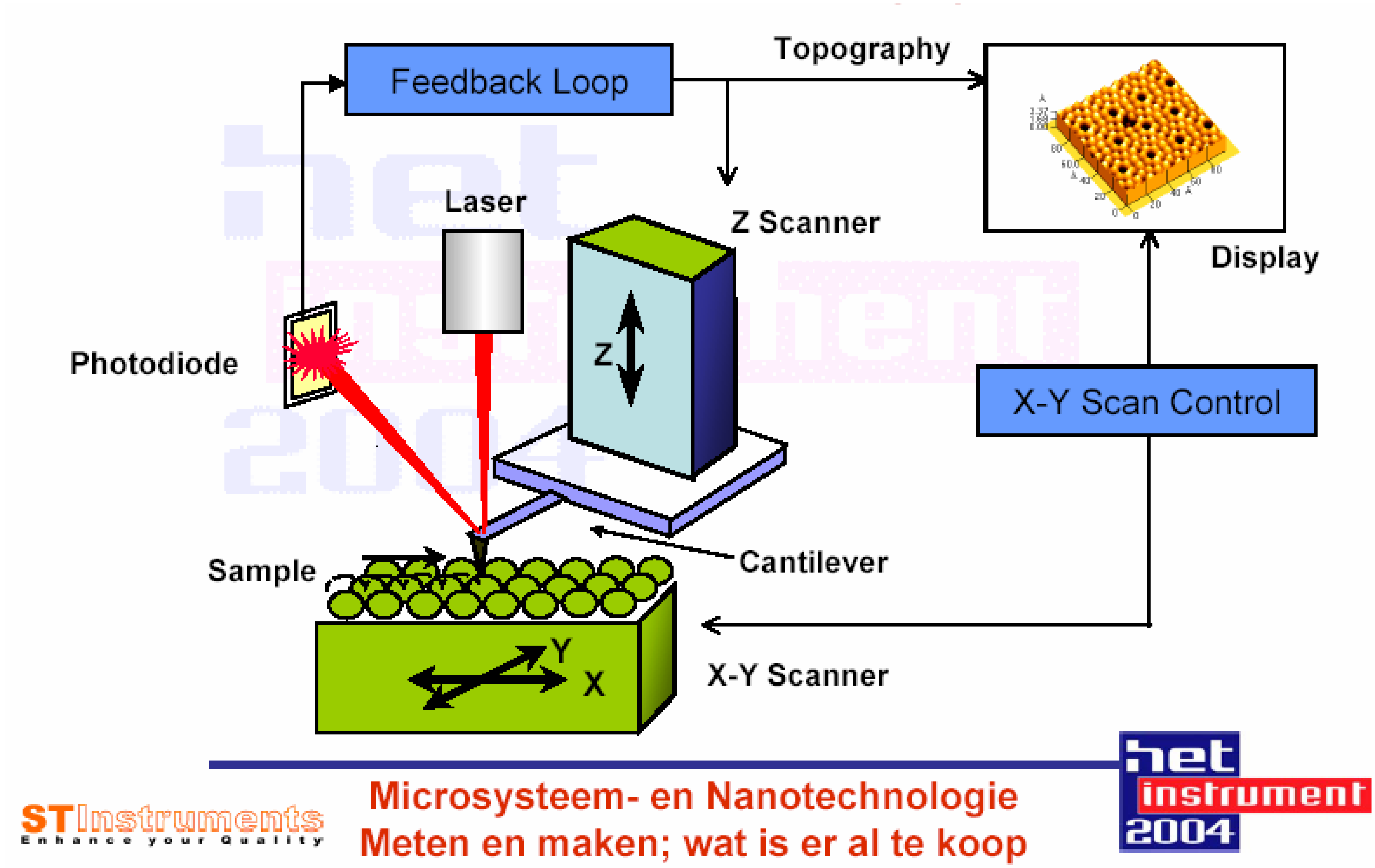






Courtesy ST Instruments

Scanning Probe Microscope (3)

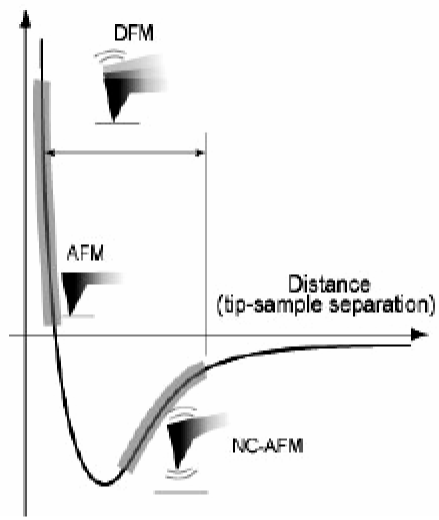


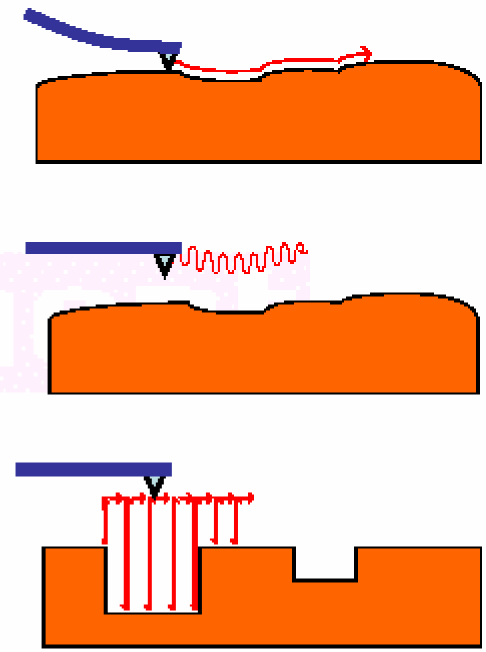
Scanning Probe Microscope (4)

AFM

There are three primary AFM modes available:

- Contact AFM
- Non contact AFM
- Dynamic AFM

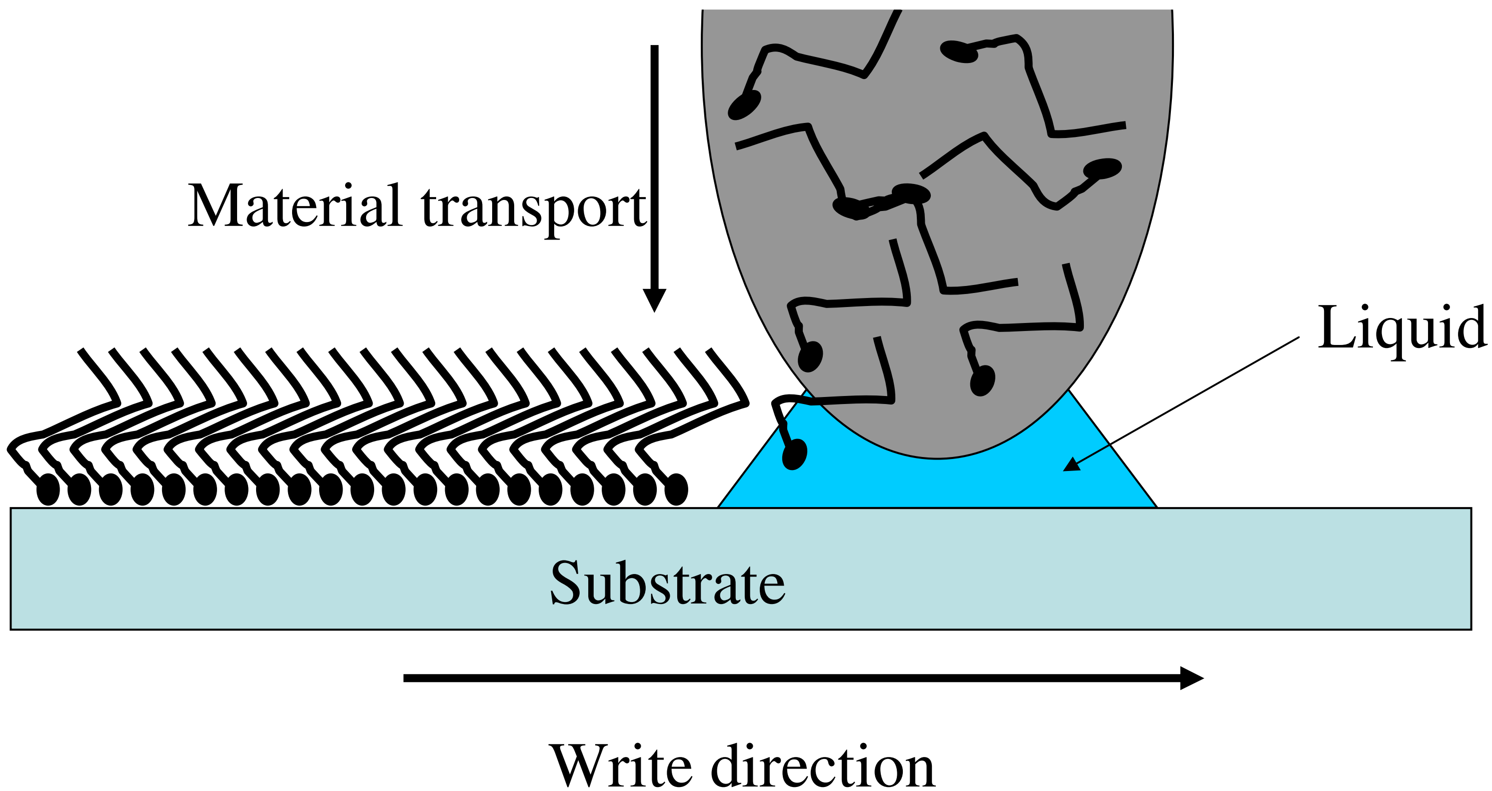




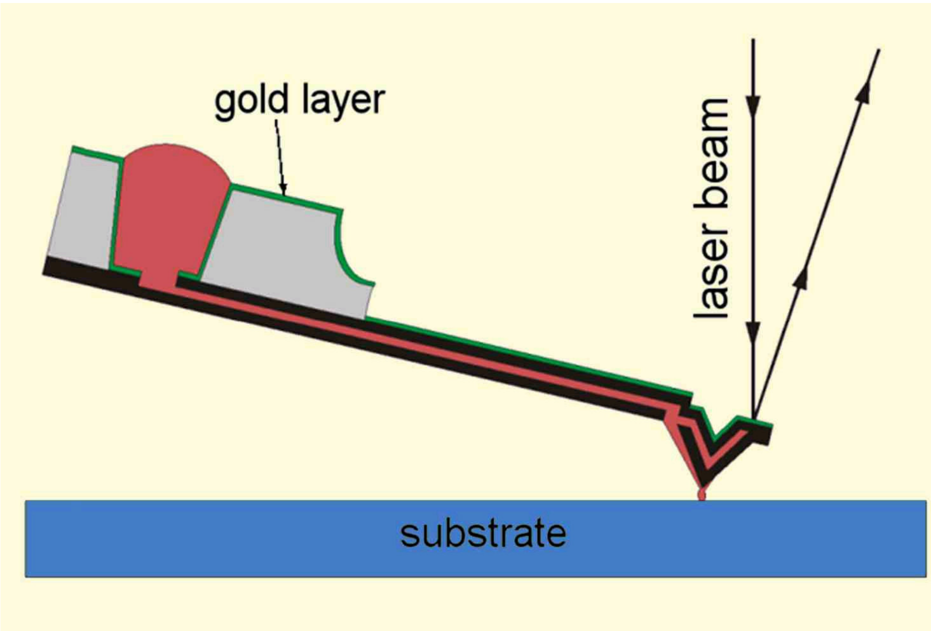
Dip pen technology (1) (Nanoink)

- Nanoink offers a SW package that transforms an AFM tool into a nano dispense unit. The achieved resolution is less then 10 nm.
- To overcome the disadvantage of the low deposition speed, the plan is to launch an array set for multiple processing. However, it is difficult to achieve a well-controlled and reliable operation for each deposition point/tip.
- Probably the most promising area for dip-pen technology will be for the deposition of bioactive materials, which can not be deposited or structured via the harsh conditions of thin film processing. (i.e. writing of proteins patterns on gold surfaces)

Dip pen technology (2) (Nanoink)

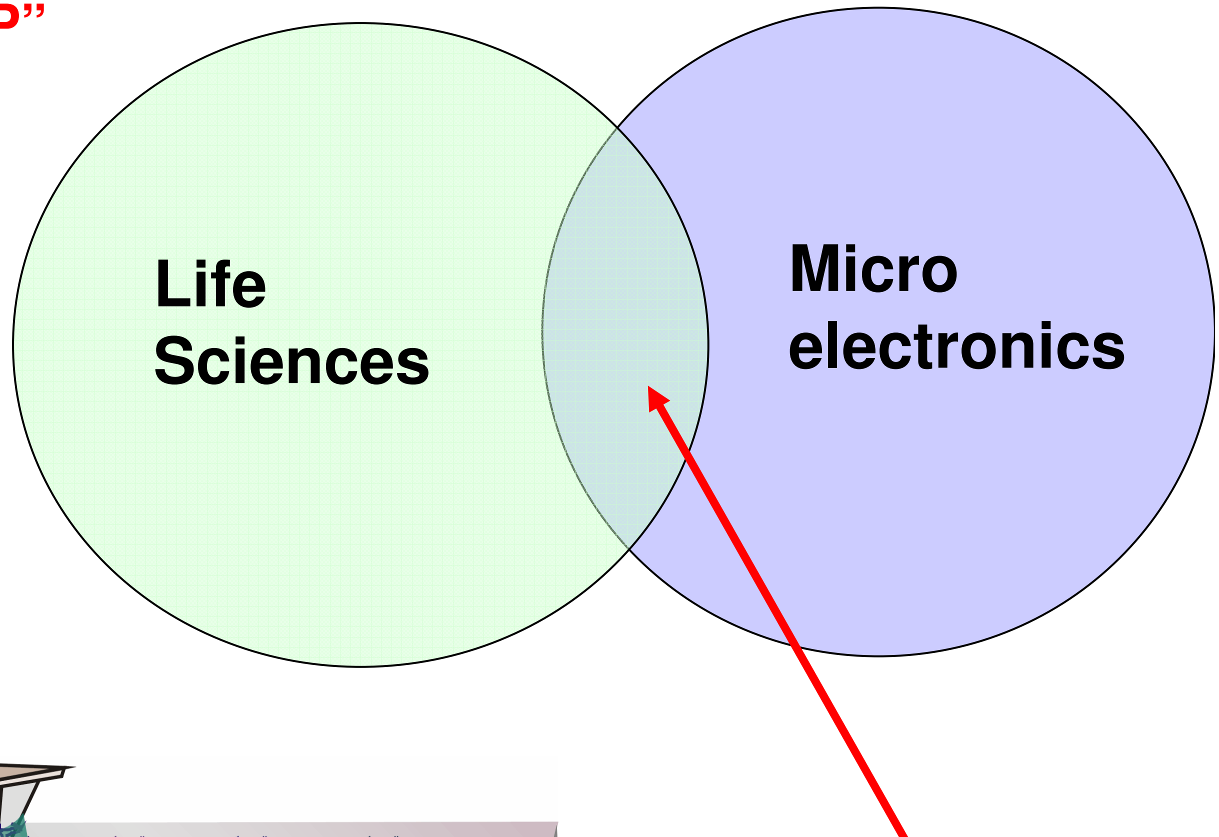


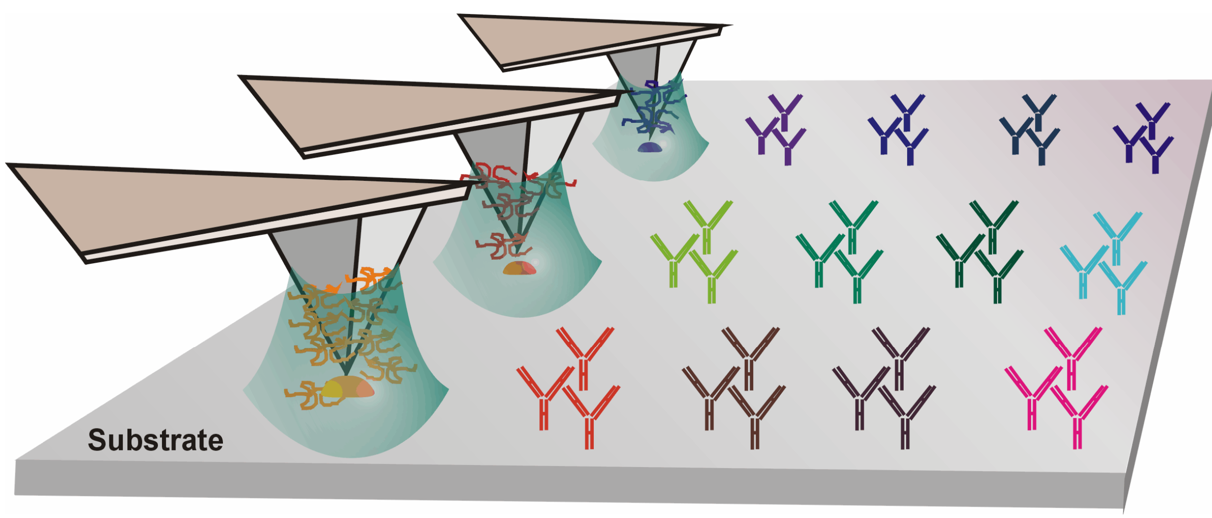
Smartip's Fountain pen



- A 'standard' silicon nitride AFM probe, with the unique feature that it has a single or dual integrated micro-fluidic channel to supply fluid to the tip.
- A new probe developed at the TST group at the MESA+ Institute for Nano-technology and commercialized by Smartip.

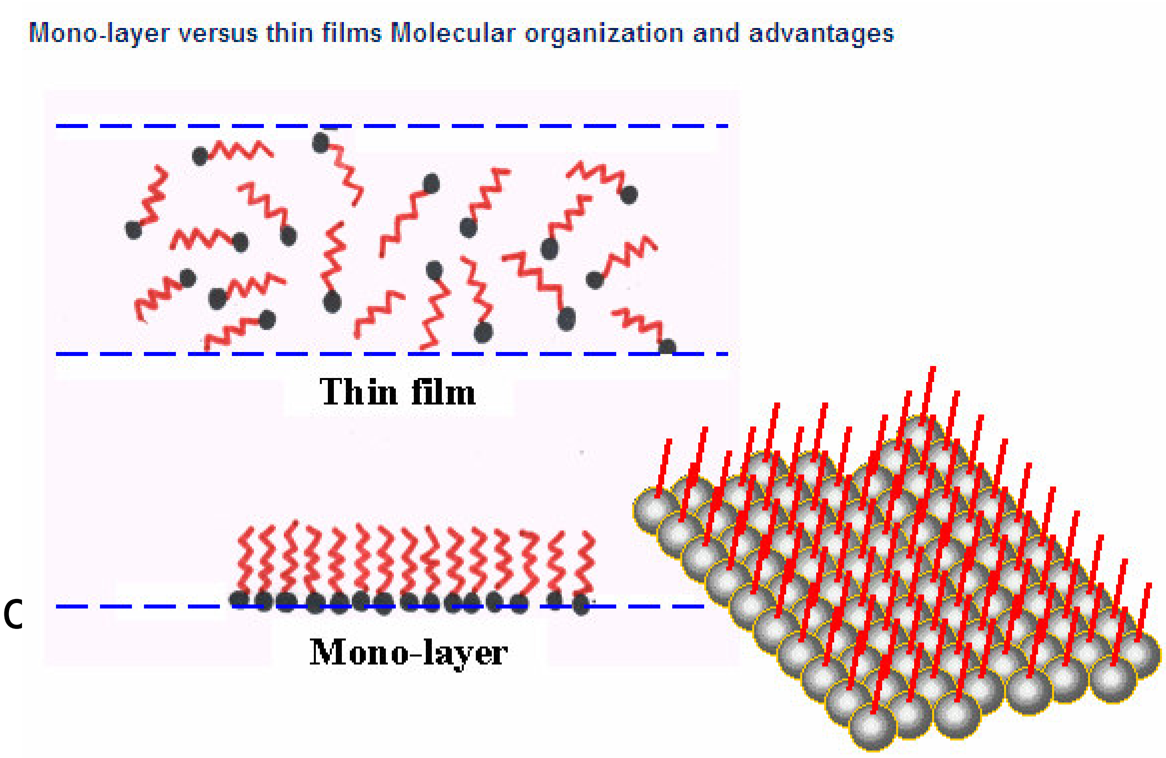
CONVERGENCE of fields from the "BOTTOM UP"





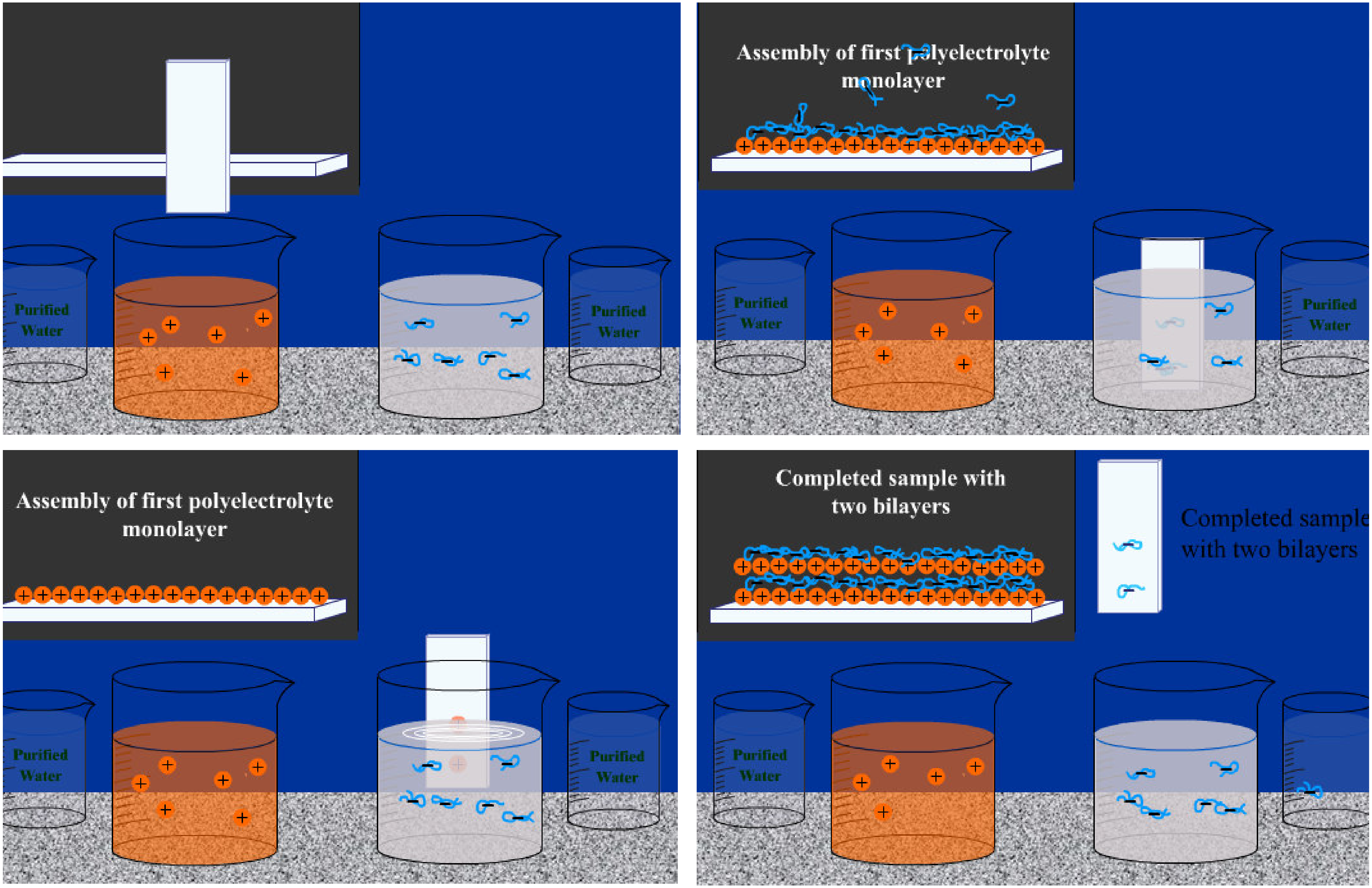
Nanometric scale assembly

- Ordered organization of matter
- High level of uniformity and reproducibility
- Large surfaces
- Continues production
- Flexibility to adapt to divers application
- Not aggressive to bio-material
- Atmospheric pressure (nc need of vacuum), room temperature and aggressive solvents not required.



Electrostatic Self Assembly (1) (nanosonic)

- Involves the dipping of a substrate into alternate aqueous solutions containing anionic and cationic materials such as:
  - complexes of polymers;
  - metal and oxide nanoclusters;
  - cage-structured molecules such as fullerenes;
  - and proteins and other biomolecules.

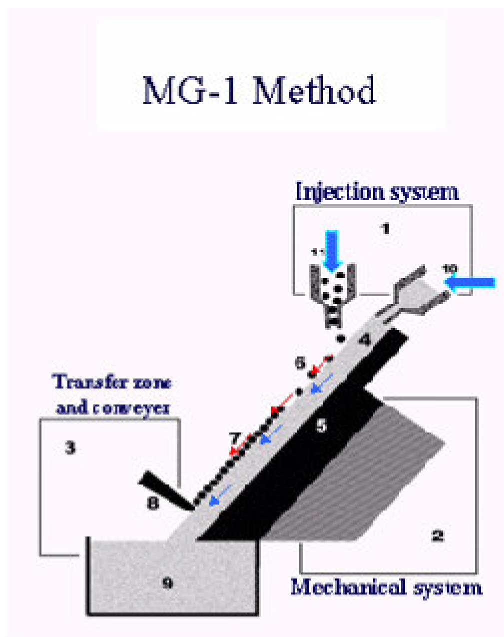

Purified
Water
Purified
Water
Assembly of first polyelectrolyte
monolayer
Purified
Water
Purified
Water
Assembly of first polyelectrolyte
monolayer
Purified
Water
Purified
Water
Completed sample with
two bilayers
Completed sample
with two bilayers
Purified
Water

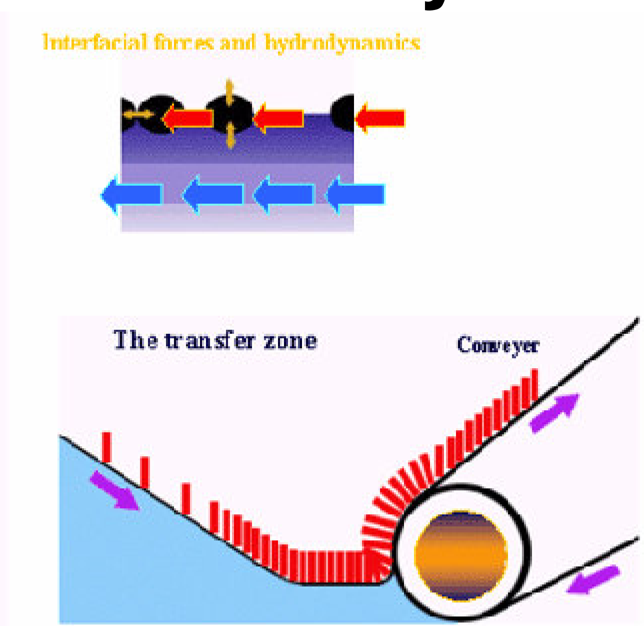
Electrostatic Self Assembly (3) (nanosonic)

- Design of these individual precursor molecules, and control of the order of the multiple molecular layers through the thickness of the film, allow control over macroscopic electrical, optical, magnetic, thermal, mechanical, and other properties, important to many engineering devices and applications.
- The nearly perfect molecular order of the individual monolayers is the net result of many individual molecules seeking local least energy configurations when adsorbed from water solutions to bond with molecules already attached at the substrate surface.

Self assembly

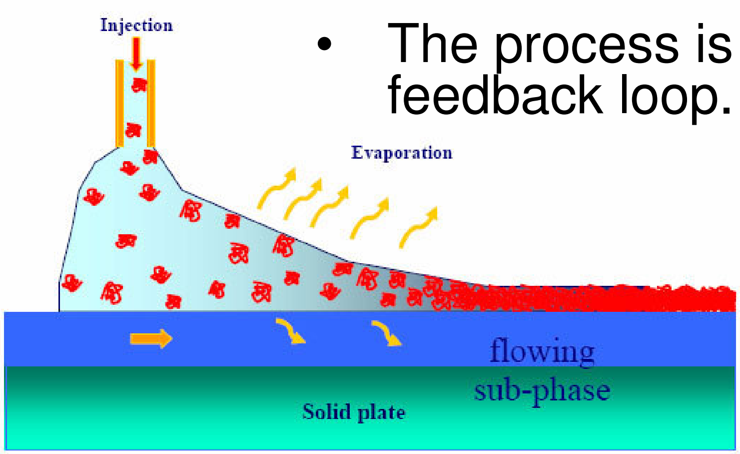
- **natural forces of gravity and hydrodynamics to facilitate the organized & structured assembly of particles**





Nanometrix' self assembly process

- The resist is layered at the molecular level on the surface of liquid.
- The coating is then applied to the wafer by lifting the wafer up through the liquid.
- The natural flatness of a liquid surface is exploited, and the force of the flow packs the molecules against one another in an orderly, linear manner.
- At the same time as the coating layer is being deposited onto the liquid and packed at the formation line, the layer is transferred from the liquid surface onto a solid substrate.
- The process is self-calibrating through a continuous feedback loop.



Summary

- Nanotubes: arc combusting
- Nanotubes: CVD (special nanotubes)
- Nanolayers: molecular selfassembly: organic molecules
- Nanoclays: sol- gel, ceramics
- Semiconductor isolating layers: ALD: (Al_2O_3, TiN, Ru, HfO_2)
- Thin film production: Nanoimprinting:
- Coatings, metal particles: Plasma processing:

Companies discussed

Lithography	ASML	www.asml.com
Nanoimprinting	MII	www.MolecularImprints.com
ALD	ASM	www.asm.com
Molecular beam epitaxy	MBE control solutions	www.mbecontrol.com
Plasma deposition	Oxford Instruments	www.oxinst.com
	Tekna	www.tekna.com
Sol-gel processing	Nanogate	www.nanogate.com
Pyrogenic processing	Adnanon	www.advanced-nano.com
Spray drying etc.	Altair	www.altairnano.com
Controlled Detonation	PlasmaChem	www.plasmachem.com
Milling	MBN	www.mbn.it
	Advanced Nano	www.advancednanotechnology.com
Low T deposition of nanotubes	CEVP	www.cevp.co.uk
Thermal Chem. Vapor Deposition	ILJIN Nanotech	www.iljinnanotech.co.kr
CO decomp. Into nanotubes)	SW NanoTechn.	www.swnano.com
Plasma spray process	inframat	www.inframat.com
Nanofiber spinning	Elmarco	www.elmarco.cz
Scanning Probe Microscopy	STinstruments	www.stinstruments.com
Dip pen technology	Nanoink	www.nanoink.net
Electrostatic Self Assembly	nanosonic	www.nanosonic.com
Self assembly process	Nanometrix	www.nanometrix.com
Fountain pen	Smartip	www.smartip.com

About enablingMNT

- The enablingMNT team utilizes its members' extensive experience and its wide network of industry contacts to offer organizations customized services in the fields of micro and nano technologies.
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